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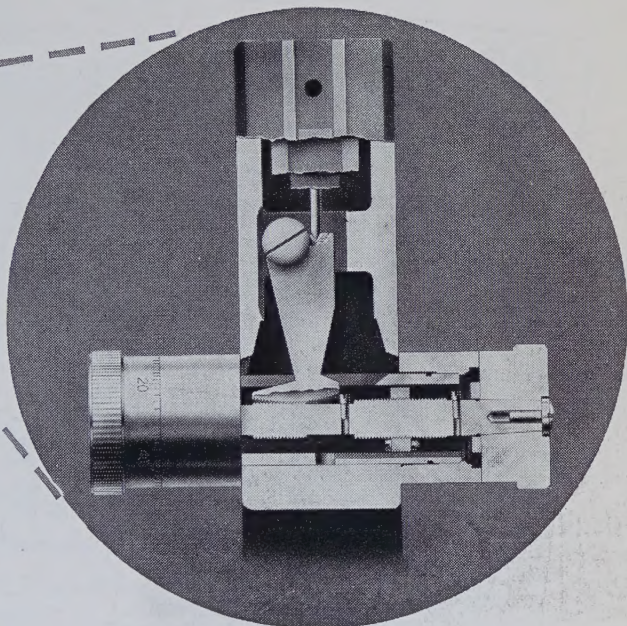
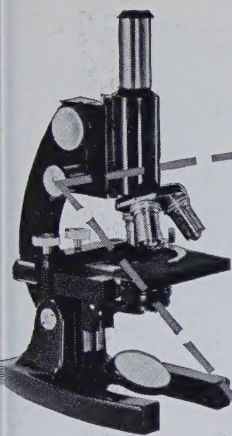
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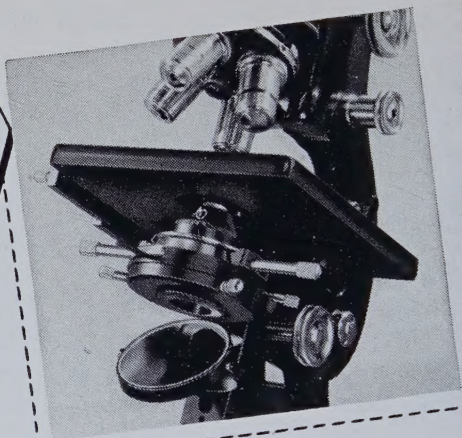


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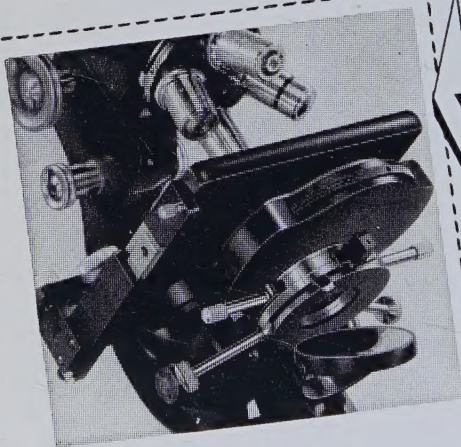
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MULTIPLE DIFFERENTIAL THERMAL ANALYSIS

PAUL F. KERR AND J. L. KULP,

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ABSTRACT

In recent years improvements in the equipment used for differential thermal analysis have been developed through work in various laboratories. As a result, the method is finding a constantly broadening application and a number of mineral groups are becoming better understood.

In this article equipment is described which is designed to further increase the utility of the method. Through the use of standard recording devices in circuit with multiple units, six differential thermal curves may be obtained simultaneously. This arrangement makes it possible to secure a large number of determinations in a short time, e.g., eighteen curves in an eight hour day. Both the equipment and a number of applications are described.

THE METHOD

Differential thermal analysis provides a useful technique for the study of specific minerals or mineral groups with distinctive heating curves. The method is suitable for both qualitative and semi-quantitative studies of the clay minerals, the hydrous oxides of iron, aluminum and manganese, the carbonates, the zeolites, and a goodly number of other minerals. In general, the method applies to substances that yield characteristic peaks in the differential thermal curves.

In this technique a dual-terminal thermocouple is employed. One terminal is inserted in an inert material which does not undergo exothermic or endothermic reaction through the temperature interval to be studied. The other is placed in the mineral or mixtures of minerals under test. With a constant heating rate a thermal reaction in the sample will be recorded as a deviation from the straight line plot of temperature difference against temperature. This deviation is dependent upon the nature of the heat change for its direction and amplitude. Peaks may be due to loss of either absorbed or lattice water, decomposition, or changes in crystal structure. They are characteristic for most thermally active minerals. Mixtures show a composite curve of the effects of the individual components in their proper proportion.

Although the original work on thermal analysis was done by Le Chatelier in 1887, it was not until the later 1930's that the method began to be used for semi-quantitative study of clay minerals. In recent years studies have been made at the National Bureau of Standards (Ewell and Insley, 1935), Massachusetts Institute of Technology (Norton, 1939), the United States Geological Survey (Alexander, Faust *et al.*, 1943), the Illinois Geological Survey (Grim and Rowland, 1942), the Bureau of Plant Industry (Hendricks *et al.*, 1946) and various Bureau of Mines research laboratories (Berkelhamer, 1945, Pask and Davies, 1945, and Speil, 1945).

Publications resulting from these studies emphasize the value of differential thermal analysis as a supplementary method coordinated with the chemical, optical, and x -ray methods in studying clay minerals. X -ray data may have certain advantages in indicating a general clay mineral group. Thermal analysis curves, on the other hand, may contribute quantitative data on mixtures not readily available from x -ray diffraction studies. Also, substitution in the clay mineral lattice is frequently more apparent in the peak shifts of thermal curves than in x -ray patterns that frequently lack suitable definition. In combination, the two methods offer a solution to many complex problems in the study of clays.

The authors wish to acknowledge the helpful criticisms of the manuscript received from R. E. Grim, Illinois Geological Survey; Ben B. Cox and Duncan McConnell, Gulf Research and Development Laboratories; M. L. Fuller, T. L. Hurst, L. D. Fetterolf and D. G. Brubaker, New Jersey Zinc Company; Robert Rowan, R. H. Sherman, Creole Petroleum Corporation; Parke A. Dickey, Carter Oil Company.

THE APPARATUS

The use of thermal analysis in the study of argillic alteration of a mineralized area, or a stratigraphic correlation problem requires the testing of hundreds of samples. This has involved a tedious laboratory procedure in the forms of apparatus described in the literature (Speil *et al.*, 1945, Norton, 1939, etc.), where a single sample is run at a time. Since each run requires several hours including cooling time, a maximum of about three samples a day may be analyzed. To overcome this difficulty as well as to provide a simultaneous comparative record, a multiple thermal analysis unit was designed (Kulp and Kerr, 1947). The various parts of the equipment were assembled late in November 1946, were placed in operation about January 1, 1947, and approximately 1,500 samples had been run by August 1, 1947.

Figure 1 shows the apparatus as set up in the Mineralogical Laboratory

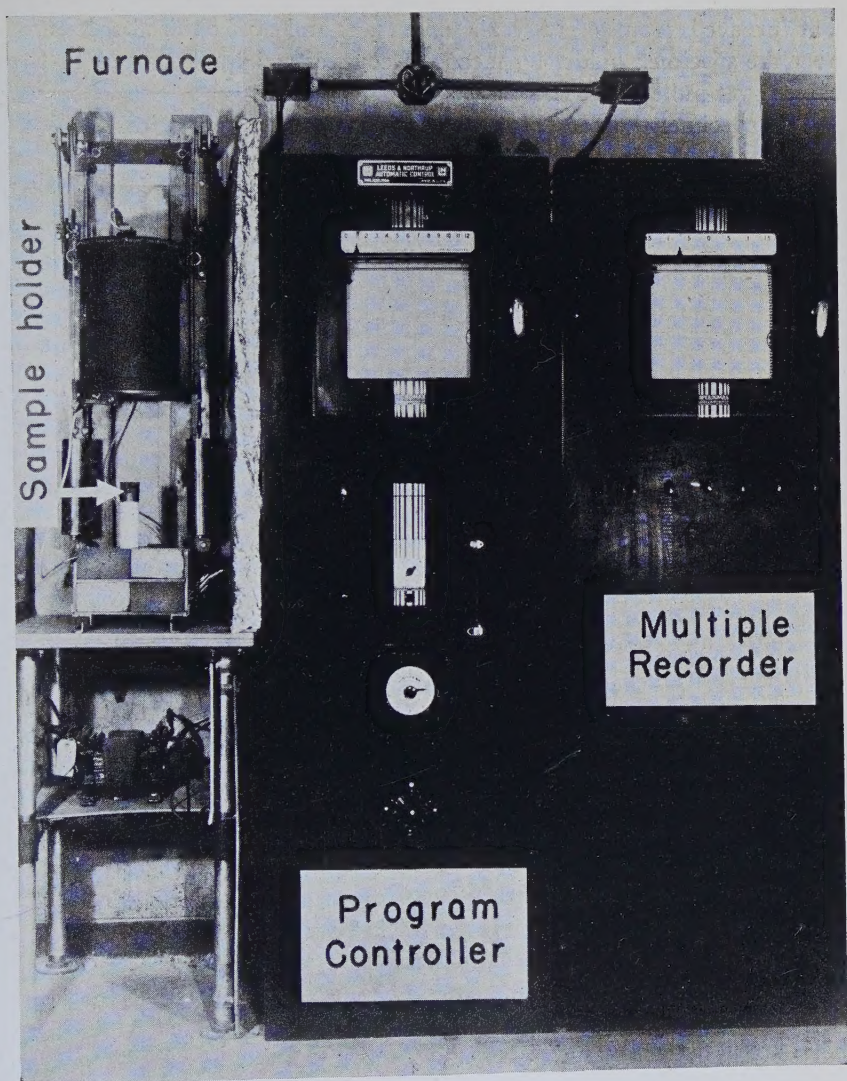


FIG. 1. Complete multiple differential thermal analysis unit.

at Columbia University. For purposes of description, the apparatus may be conveniently divided into four parts: the furnace, the sample holder, the program controller, and the multi-recorder.

The furnace is a Hoskins 305 electrical resistance furnace into which an alundum tube ($1\frac{3}{4}$ " i.d. $\times$ 12" $\times$ $\frac{3}{16}$ " wall) is inserted in order to diffuse the heat and to insulate the metal specimen holder from the heater coils.

The furnace is mounted vertically on a track and can be raised or lowered over the specimen holder by means of counter weights attached to two cables over pulleys.

The specimen holder (Fig. 2) is drilled from a cylindrical block of chrome nickel steel $1\frac{5}{8}$ " outside diameter and 1" height. Both pure nickel and chrome nickel steel have been used but the latter has similar heat conductivity and is less subject to scaling. The six samples to be tested

PLAN VIEW

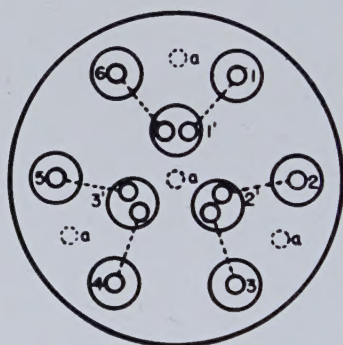


FIG. 2. Nickel specimen holder.

are loaded in the outer holes numbered 1 to 6, while the inner holes 1', 2', 3' are used for inert material which is ordinarily purified alundum, manufactured by the Norton Company. The dashed lines indicate the connections between the two terminals of the chromel-alumel differential thermocouples. Thus one hole containing alundum is sufficient for the inert side of two differential couples. Chromel-alumel couples, BXS 22, were used for maximum e.m.f. generation and were found to be substantial. The dots "a" indicate the positions of the temperature recording thermocouples. The terminals of these couples are adjusted to the same height as the differential couples in the samples. The sample and alundum holes are $\frac{1}{4}$ " diameter and $\frac{3}{8}$ " deep.

The chrome-nickel steel block is supported by an alundum tube ($1\frac{1}{8}$ " i.d. $\times$ $6\frac{1}{2}$ " $\times$ $\frac{1}{4}$ " wall), and supports a cylindrical cover of solid nickel $\frac{1}{4}$ " thick which is placed on the block to shield the samples from direct radiation. Two complete units of sample holder and thermocouples were prepared. Thus if a break occurs in one thermocouple, the entire unit may be recovered without delay and a replacement connected. The next run may thus be carried out without loss of time for repairs.

The program controller is a special Leeds and Northrup "micromax" which is connected to one of the four possible temperature recording

thermocouples by way of a rotary selector switch. This unit is rated to raise or lower the temperature of the sample at any desired rate from 0 to 50° C. per minute. It will also automatically hold the sample at any desired temperature when that temperature is reached. The pen record indicates the temperature of the sample. The controller, when properly adjusted, gives a linear heating curve.

The recorder for the differential thermocouples is a Leeds and Northrup "speedomax," six point, high speed, high sensitivity electronic recorder with a maximum range of 3 millivolts. The chart of this recorder is synchronized with the chart containing the temperature record on the program controller. This recorder is sensitive to 0.1° C. differential temperature which, with the present specimen holder, gives a peak one centimeter in amplitude for the alpha-beta quartz change. Experimentation on increasing sensitivity with accessory devices is in progress. However, it should be pointed out that beyond a certain limit of sensitivity, thermal gradients, geometry, thermal couple defects, and other unknown factors cause prohibitive irregularities in the base line. The present equipment yields curves that are reproducible to a degree or so in peak temperature and to five per cent of the peak amplitude under normal conditions.

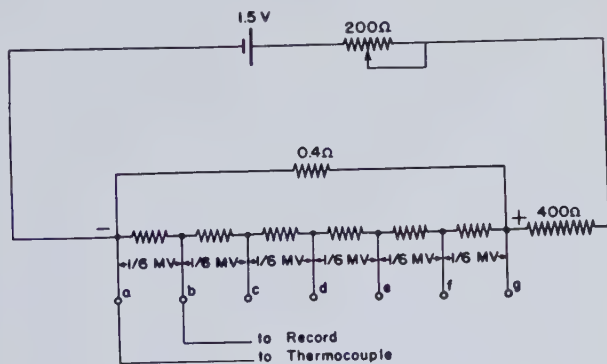


FIG. 3. Potentiometer circuit for spreading records. The unit is placed in series with one head of each thermocouple. The desired position for each couple is achieved by connecting across appropriate terminals from "a" to "g." In the diagram, connection on "a" and "b" would add a constant $\frac{1}{5}$ mv to the base line of the differential thermal curve.

Since all the differential thermocouples print at zero millivolts where there is no reaction taking place, it is desirable to spread the six records. This is done by a simple potentiometer circuit (Fig. 3) which places the base line of each record about one-sixth of a millivolt from its nearest neighbors. The exact separation desired is achieved by adjusting a 200 ohm resistance in the battery circuit. Also it is desirable to have certain

sensitivity scales available since some of the reaction minerals: alunite, jarosite, kaolinite, carbonates, etc., may extend beyond the chart on high sensitivity. Since this type of recorder measures the e.m.f. of the thermocouple, a simple voltage divider with proportionate resistances is efficient for obtaining one-half, one-third, or any other predetermined fraction of the generated e.m.f.

Finally, there are two solenoid pens in series, one attached to the edge of each recorder. By means of a button switch, the solenoids are simultaneously activated, thus marking both records at the same time. Since the temperature at that instant can be read from the program controller record, the temperature of the six records is also known and can be written on the multi-record chart at the completion of the run.

The advantages of this equipment are worthy of note. One of the greatest is the multiple record feature, by means of which with three runs eighteen samples may be tested conveniently in an eight hour day. Also significant is the reduction in the number of potential variables in using six samples under the same heating conditions. This is important when runs of quantitative mixtures are compared. The unit is compact, it does not require a darkened room for operation as in the photographic recording methods, and the results are immediately observable. The chief disadvantage lies in the necessity for applying minor corrections to each curve.

PROCEDURE

The samples to be tested by the differential thermal analysis apparatus are passed through a 50 mesh screen and packed to finger tightness around the differential thermocouple. No pretreatment is given for an ordinary run. It has been found by experimentation, as reported by others, that any attempt to attain equilibrium with a specified humidity merely alters the initial absorbed water peaks (100–200 degrees C.) whose amplitudes are usually not used for quantitative analysis. Ordinarily weighing has been found to be unnecessary and reproducible curves may be obtained for the same pure substance with finger-tight packing with a close fitting metal plunger to a constant level. In special cases attention must be given to the problems of particle size, weight, and humidity.

After the samples are loaded, the cover placed on the specimen holder, and the two charts synchronized, the furnace is started. The heating rate has been standardized at 12 degrees per minute since this gives sensitive control, produces adequately sharp peaks, and is close to the heating rate used by a number of other workers in this field. The record is made from 100 to 1050 degrees Centigrade. At the beginning and end of the run the button switch activating the solenoid pens is pushed, thus fixing the

temperature on the multiple differential thermocouple record. When 1050 degrees C. is reached, the furnace is raised from the specimen holder and the samples removed by compressed air while they are still hot. This procedure prevents "caking" which occurs in certain specimens upon cooling.

The temperature thermocouples are calibrated and recalibrated occasionally with the alpha-beta quartz change. It has been found after trying many different thermocouples that the quartz inversion peak occurs on a differential curve within 5° of 579° C. (This is higher than the equilibrium value.) The reproducibility has also been observed with the standard Georgia kaolinite endothermic and exothermic peaks. Since this is consistent with data in the literature and since the change in peak temperature of pure hydrous minerals may easily vary 5° C., more precise calibration has been considered unnecessary. Different sample blocks, thermocouples, and furnace windings produce no change in peak temperature greater than 5° C.

Although the thermocouples are made as similar as possible and adjusted to approximately the same heights in the sample holes, the sensitivity varies slightly. Therefore, after replacement of one specimen holder and the corresponding thermocouples by another, the first run is usually made with standard Georgia kaolinite in all sample holes. This indicates the relative sensitivity of the various thermocouples. It has been found that these relative sensitivities remain essentially constant for the life of the thermocouples unless the height of the thermocouple is changed due to rough handling.

All curves included in this description are based on the same sensitivity for direct comparison. It has been found convenient to plot the differential thermal curve so that an exothermic peak is upward while an endothermic reaction is represented by a deviation downward from the base line curve.

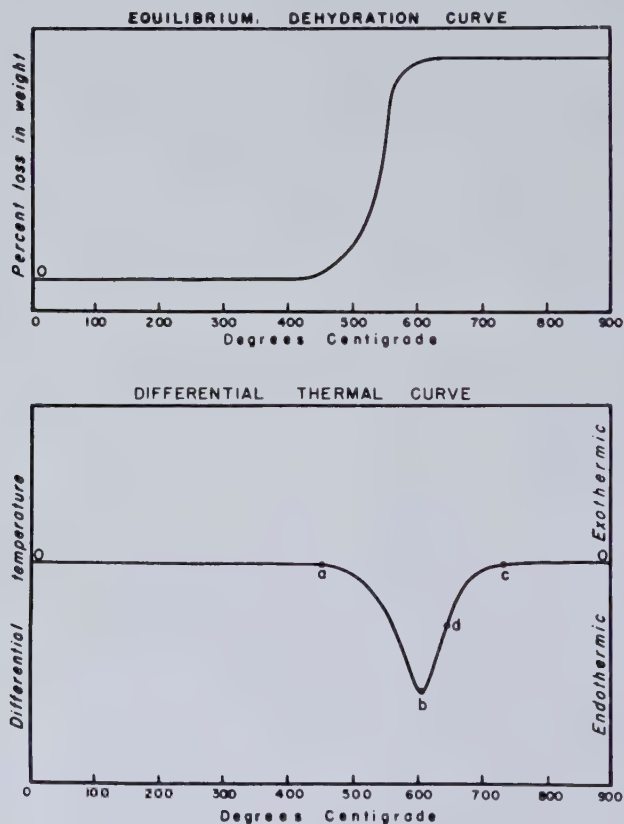
THEORY

The theory of differential thermal analysis has been presented by Speil (1945). The following account, modified and corrected,* is included to aid in introducing the present studies.

Figure 4 compares two methods of dehydrating a clay mineral. The static method produces the equilibrium dehydration curve while the dynamic method gives the differential thermal curve. In the first instance, the sample is held at each successively higher temperature until it has reached equilibrium. In the latter, the sample is heated at a constant rate,

* Error in Speil's derivation pointed out by Dr. D. G. Brubaker, N. J. Zinc Co., Palmerton, Pa.

thus extending the dehydration over a longer temperature range. Since the thermal curve is a differential function, it depends only on those effects that do not occur simultaneously and equally in the specimen and the inert material. Hence, there are only two thermal effects to consider, the differential flow of heat from the block to or from the thermocouple in the center of the sample and the heat of the thermal reaction. The differential thermal curve of Fig. 4 represents an endothermic reaction. Below



THEORETICAL THERMAL CURVES

FIG. 4. Theoretical thermal curves.

temperature "a," the heat inflow to both thermocouples, sample and inert material, is the same and no difference in temperature is recorded. At "a" the reaction starts absorbing heat from its surroundings making the sample couple cooler than the alundum couple. This effect increases until at "b" the rate of heat absorption by the chemical reaction equals

the rate of differential heat conductivity into the clay specimen. The rate of heat absorption then continues to decrease more rapidly than the inflow of heat from the block. At some point "d" between "b" and "c," the reaction ceases. However, since this point cannot be established exactly, "a" and "c" are usually chosen as limits.

Under static conditions the heat effect would cause a rise in temperature ΔT_s , of a specimen given by:

$$(1) \quad \Delta T_s = \frac{M(\Delta H)}{M_0 C}$$

where M = The mass of reactive mineral,
 H = Specific heat of reaction,
 M_0 = Total mass of specimen, and
 C = Mean specific heat of specimen.

However, the heat flow from the nickel block towards the centers of the two sample cavities must be taken into account.

For any point between "a" and "c," the simplified equation describing the changes in heat content of the thermally active constituent is:

$$(2) \quad M \int_a^x \frac{dH}{dt} dt + gk \int_a^x (T_0 - T) dt = M_0 C (T - T_a)$$

(A)
(B)
(C)

for the inert sample:

$$(3) \quad g \cdot k' \int_a^x (T_0 - T') dt = M_0' C' (T' - T_a')$$

(B)'
(C)'

where t = Time,

M_0 = Total mass of test specimen,
 M_0' = Total mass of alundum,
 C = Mean specific heat of test specimen,
 C' = Mean specific heat of alundum,
 k = Conductivity of the specimen,
 k' = Conductivity of the alundum,
 g = Geometrical shape constant,
 T_0 = Temperature of the nickel block,
 T_a = Temperature at center of sample at Time $T = a$,
 T_a' = Temperature at center of alundum at Time $T = a$,
 T = Temperature at center of sample,
 T' = Temperature at center of alundum

Factor (A) defines the quantity of heat added to or subtracted from the test specimen due to reaction. In an exothermic reaction dH/dt is positive. Factor (B)* defines the quantity of heat absorbed by the specimen. $(A) + (B) = (C)$ because at any point x along the differential

* The temperature gradient in the chrome nickel steel can be neglected as the thermal conductivity of the metal is so much greater than that of the refractory sample to be tested.

thermal curve, the amount of heat used in raising the temperature of the specimen must equal the amount brought in by flow from the metal block plus the amount added or subtracted by the reaction.

In the inert sample factor (A) does not exist, hence the heat which flows in (B)' must equal the heat used in raising the temperature of the specimen (C).'

Letting

$$C' = C + \Delta C$$

and

$$k' = k + \Delta k.$$

Also in the experimental procedure $M_0 = M_0'$ within the error of measurement. Subtracting (3) from (2) and rearranging gives:

$$\begin{aligned} (4) \quad M \int_a^x \frac{dH}{dt} dt + gk \int_a^x (T' - T) dt - g\Delta K \int_a^x (T_0 - T') dt \\ = M_0 \{ C[(T - T_a) - (T' - T_a')] - \Delta C[T' - T_a'] \} \\ = M_0 \{ D[(T - T') - (T_a - T_a')] - \Delta C[T' - T_a'] \}. \end{aligned}$$

As $T' - T = T =$ temperature indicated by the differential thermocouple, the equation can be considerably simplified by assuming that the terms containing $(T_0 - T')$, C , and K are small in comparison with other terms. By using "a" and "c" as integration limits:

$$(5) \quad M \int_a^c \frac{dH}{dt} dt + g \cdot k \int_a^c \Delta T dt = M_0 C [(T_c - T_c') - (T_a - T_a')]$$

but to a close approximations*

$$(T_a - T_a') = (T_c - T_c')$$

and

$$M \int_a^c \frac{dH}{dt} dt = M\Delta H,$$

the total heat of reaction

$$(6) \quad \text{so,} \quad \frac{M(\Delta H)}{g \cdot k} = \int_a^c \Delta T dt.$$

The last expression is proportional to the area enclosed by a straight line from "a" to "c," and the curve "abc," if the deviation from the base line is a linear function of the differential temperature. It is proportional, therefore, to the percentage of reacting material in a given weight of sample. This forms the basis for the quantitative use of differential thermal analysis. The linear relationship holds reasonably well. More

* $(T_a - T_a')$ and $(T_c - T_c')$ will equal zero for specimen holders in which the test and alundum holes are symmetrically spaced relative to the heat source. In the present concentric type of spacing $(T_a - T_a') = (T_c - T_c')$ within the error of measurement.

exact determinations of comparatively simple systems can be made by running known mixtures and preparing a calibration curve of area (vs.) percentage of each component.

The above derivation neglects the differential terms and the temperature gradient in the sample. It shows that the area under the curve is a measure of the total heat effect. The area is also considered independent of the specific heat. This factor, however, actually does affect the shape of the peak and may change the area slightly. For many purposes the approximate relationships are sufficient.

Qualitative Applications

The various clay minerals yield sufficiently different peaks to make the differential thermal analysis method particularly useful. When a specimen is relatively pure, preliminary identification by thermal curves is frequently comparatively simple. In addition, two component mixtures are often resolved and even three component mixtures at times. If, however, mixtures become too complex, only one or possibly two of the major components may be identified.

The multiple thermal analysis apparatus makes possible a rapid widespread survey of the groups of minerals that can be identified by this procedure. The thermal curves given here are representative of the results obtainable. It is believed that they agree substantially with those of the other workers on record. Illustrative curves are shown for the kaolinite and montmorillonite groups along with the hydrous oxides of aluminum and iron, some sulfates and carbonates.

The kaolin minerals, Figs. 5, 6, 7, are characterized by a large endothermic peak ranging from 550° C. to 700° C., due to the decomposition of the kaolinite lattice into amorphous silica and alumina, and a sharp exothermic peak at 980° C. caused by the recrystallization of amorphous alumina to gamma alumina.

Thermal curves of dickite from several localities are shown in Fig. 5. A number of the samples illustrated have been studied in connection with other investigations to such an extent that they may be considered representative of this clay mineral. The sample from Red Mountain, Colorado, was ground and curves were run to compare 100–200 mesh, 200–300 mesh and smaller than 300 mesh material. It is interesting to note that an ordinary specimen from St. Peter's Dome yields a curve similar in character to Red Mountain dickite ground to minus 300 mesh. It is evident that particle size is a factor to be considered.

As the degree of orderliness in the superposition of the kaolin layers decreases from dickite through kaolinite to halloysite, the endothermic peak shifts downward in temperature, indicating less stability of the

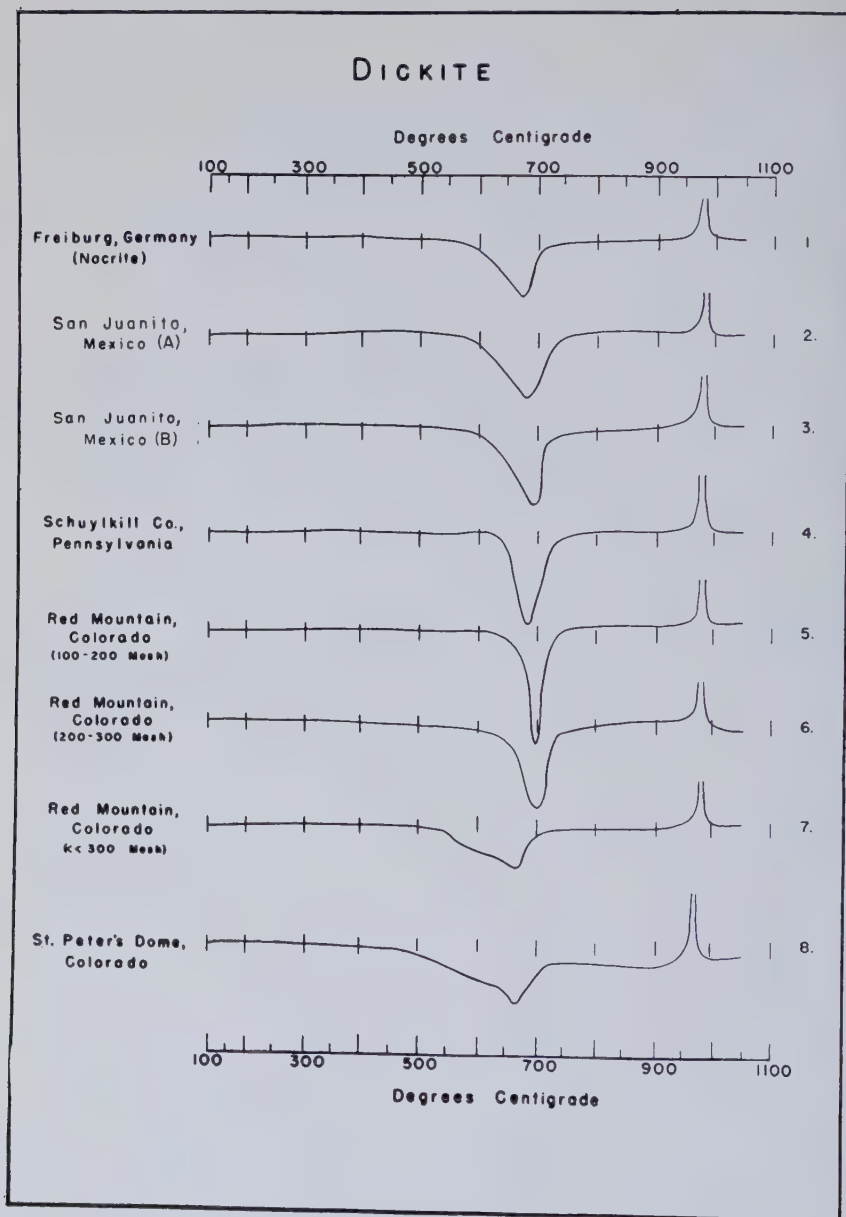


FIG. 5. Thermal curves of dickite from several localities. Curves 5, 6 and 7 indicate the effect of grinding on dickite from Red Mountain, Colorado.

KAOLINITE

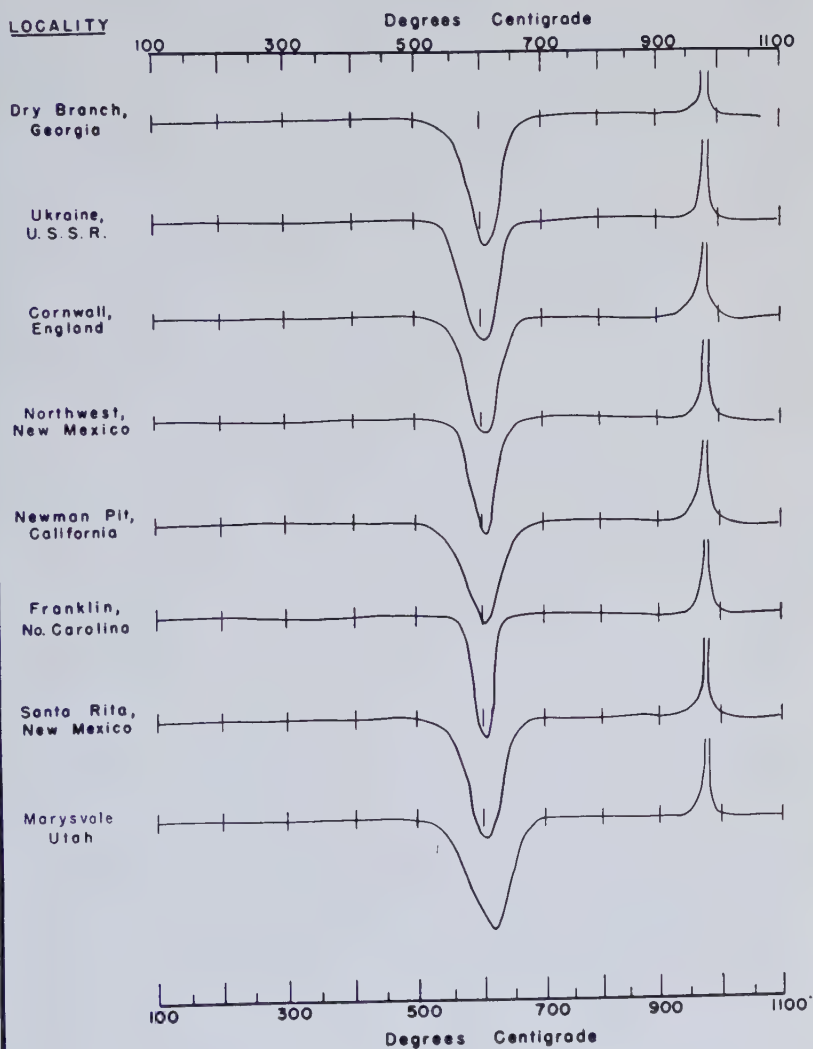


FIG. 6. Thermal curves of 8 specimens of kaolinite from different localities, all similar in character.

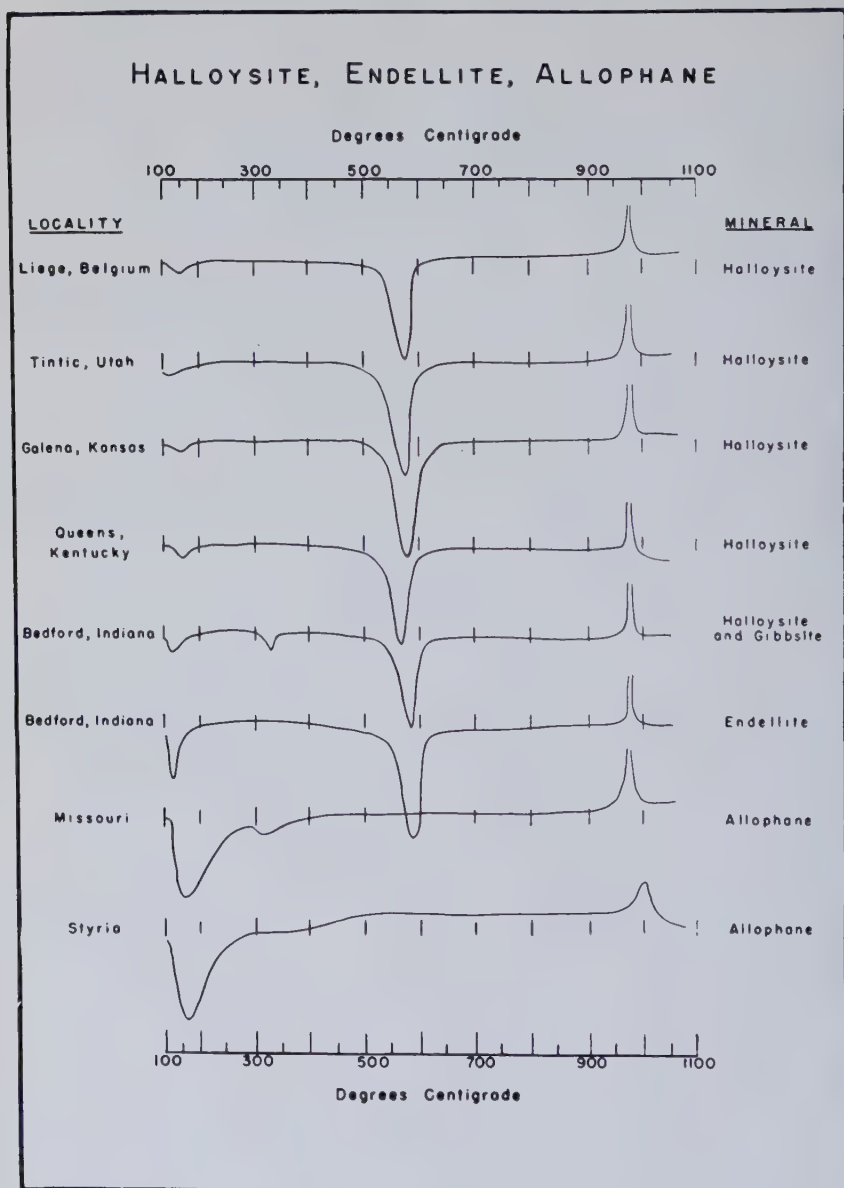


FIG. 7. Thermal curves of halloysite, halloysite mixed with some gibbsite, endellite and allophane.

lattice. The disorder reaches a sufficient extent in the case of halloysite to permit absorption of some water between the lattice layers. This accounts for the minor endothermic peak at about 150° C. This peak is greatly enlarged in the case of endellite, the more hydrous form of halloysite. Endellite yields water to form halloysite under 100° C. Allophane has been considered in the kaolin group (Speil, 1945) presumably because of the sharp 980° C. exothermic peak corresponding to the formation of the gamma alumina, and a rough agreement of chemical analyses. The kaolin group is best delimited by the unique lattice type which is observed in nacrite, dickite, kaolinite, and halloysite.

Figure 6 contains a series of kaolinite curves. The variations in the shape of the endothermic peaks are probably due to differences in clay mineral particle size distribution. The samples with a narrow range of particle size appear to give the sharpest peaks. Since the total heat evolved is dependent only on the concentration of reactive molecules present around the thermocouple, the area under the curve should be roughly constant. It is evident from the set of curves that the differences in the shapes of the curves are not great and, hence, the amplitudes are essentially the same. The specimens from Dry Branch, Ga.; Cornwall, England; Newman Pit, California; Franklin, N. C.; and Santa Rita, N. M., give characteristic kaolinite x-ray patterns. The Georgia material was used as a standard for comparison.

The samples were prepared by passing the kaolinite through a 50 mesh screen. One specimen (not shown) of Georgia kaolin "fines" obtained by gravity separation exhibits a kaolinite curve with an endothermic peak depressed slightly and lowered in temperature about ten degrees. The 980° C. exothermic peak of this material also shows a slight shift to lower temperatures. This probably is due to the finer particle size material which is in a less stable state with a correspondingly lower temperature of recrystallization.

A private communication from R. E. Grim indicates that some kaolinite samples give curves with upward swings in the thermal record between the large endothermic reaction and the final exothermic reaction. Also these same kaolinite samples give a slight endothermic dip just before the final exothermic peak. Of the limited number of kaolinite curves shown in Fig. 6, only the Marysville, Utah and Northwest, N. M., samples indicate the slight endothermic peak mentioned above.

Figure 7 contains typical thermal curves of halloysite, endellite, and allophane. The first four halloysite samples are believed to be of high purity. X-ray diffraction photographs have been obtained from these four specimens and display the lines of halloysite. The thermal curves are similar to those of kaolinite with two significant differences, the small

endothermic peak at 150° C. due to adsorbed water, and the shift in the main endothermic peak to about 570° C. Grim (1942) claims halloysite does not have a lower temperature for the main endothermic peak than kaolinite. Other workers (Speil, Berkelhamer, Pask and Davies, Norton) show evidence of the lower temperature of the halloysite peak. In this laboratory eight typical halloysite specimens yield endothermic peaks at 575° C. $\pm$ 10° C. On the other hand, the kaolinite samples examined show endothermic peaks at 600° C. or above. This difference is well above the limits of experimental error. One possible complication is noted in the case of very fine kaolinite. Here the 605° C. peak is shifted downward toward the halloysite peak. However, the fine kaolinite does not have as low an endothermic peak as does halloysite, the amplitude and the shape of the 600° C. peak is altered, and the 980° C. exothermic peak is shifted down the temperature scale.

These data are not sufficiently conclusive to establish the range of the endothermic peak of kaolinite. The structure variations in halloysite and kaolinite and the correlation with the thermal phenomena require further investigation.

The halloysite from Bedford, Indiana, contains a small amount of gibbsite. The endellite from Bedford is typical, showing the halloysite curve with a greatly increased low temperature endothermic peak. The last two curves in Fig. 7 are typical of allophane.

Figure 8 contains the thermal curves of certain 3-layer lattice minerals. Montmorillonite furnishes a broad classification for a certain crystal structure, but with wide substitution possibilities in the lattice. An excellent paper by Ross and Hendricks (1945) has contributed to the clarification of this group. The thermal curve of the Polkville, Miss., material exhibits the previously recognized low temperature doublet, the two high temperature endothermic peaks and the final high temperature exothermic peak. The amplitude of the doublet is dependent to a large extent on the humidity conditions before thermal analysis. Hendricks *et al.* (1940) pointed out that the shape of these peaks was due to the quantity of adsorbed water and the type of adsorbed cation between the 3-layer units. The high temperature endothermic peaks occur variably between the limits of 550° C. and 1000° C. This is probably to be attributed to substitution within the layer itself. The temperature of the peaks has not as yet been correlated with chemical analysis. This is now being investigated. The high temperature exothermic peak is dependent in part on the substitution of iron for aluminum within the layer. The substitutions in the montmorillonite lattice are perhaps more apparent from the shifts in the thermal curve peaks than the shifts in the lines of the diffuse x -ray diffraction patterns.

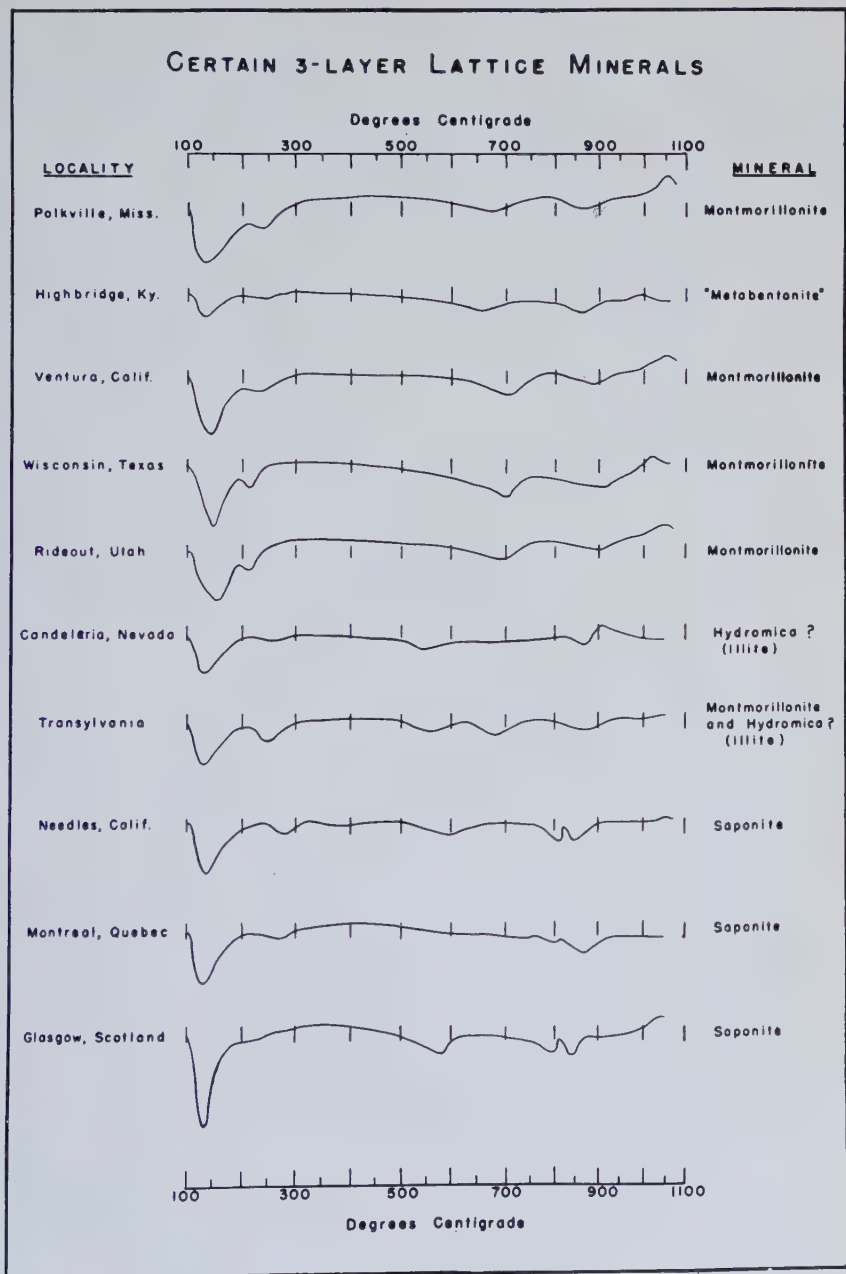


FIG. 8. Several thermal curves of montmorillonite, saponite and examples of hydromica (metabentonite and illite) are illustrated.

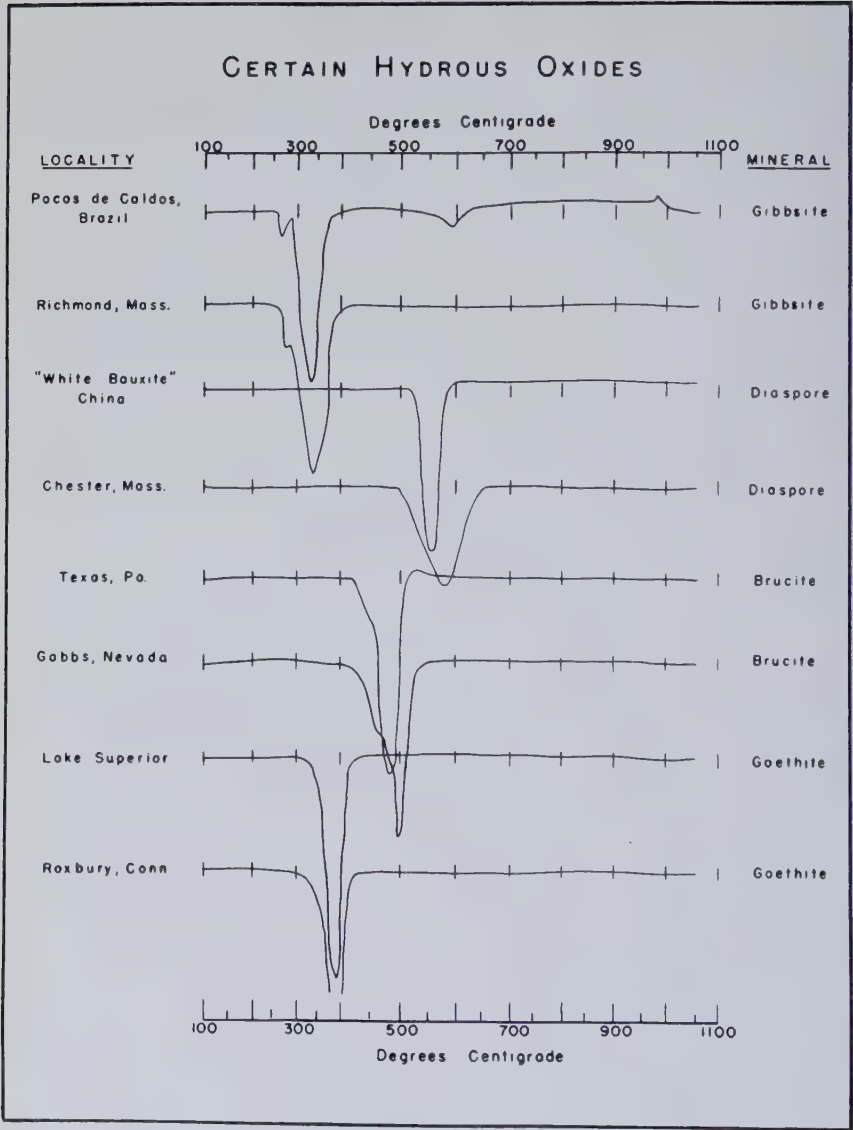


Fig. 9. Curves of gibbsite, diaspore, brucite and goethite are shown for comparison.

The curves of specimens from Ventura, California; Wisconsin, Texas; and Rideout, Utah, are typical of montmorillonite. The "metabentonite" from Highbridge, Ky., contains potash but the thermal curve indicates a material more like montmorillonite than hydromica or illite where the first high temperature endothermic peak occurs at 600° C. or lower. The specimen from Candelaria, Nevada, shows a thermal curve more characteristic of hydromica while that from Transylvania appears to be a mixture of montmorillonite and hydromica (illite).

The last three curves of this set are from specimens labelled "saponite," the high magnesium montmorillonite clay. This mineral shows a distinct double peak in the neighborhood of 800–850° C. All of these specimens give saponite x -ray patterns.

Figure 9 shows thermal curves of gibbsite, diaspora, brucite, and goethite. All of these specimens were checked by x -ray diffraction. The curves for gibbsite agree with those in the literature which show the main endothermic peak to occur from 330–350° C. Although Speil's (1945) sample does not show the lower temperature minor endothermic peak, the others do. This may be assumed as due to the high purity of Speil's sample. Pask and Davies (1945) ascribe the initial minor endothermic peak to clachite. The specimen from Poços de Caldos appears to contain a small amount of kaolinite.

The diaspora labelled "white bauxite" from China is apparently unusually uniform in grain size. It powders readily on crushing the sample, making grinding unnecessary. Conversely, the coarsely crystalline diaspora from Chester, Mass., requires considerable grinding. The resulting material evidently has a large grain size distribution as indicated by the shape of the curve. Apparently, both of these specimens are of high purity.

Typical well-crystallized brucite specimens from Texas, Pa., and Gabbs, Nevada, give thermal curves which correspond. Similarly specimens of goethite from the Lake Superior copper district and Roxbury, Conn., agree with each other.

Curves for alunite and jarosite are shown in Fig. 10. These minerals display prominent peaks that are distinctive and can be detected in the presence of foreign materials. The specimens from Bulledehah and Barranca probably contain inert impurities such as sericite which has depressed the peaks. Those from Santa Rita, Hyagoken, Los Lamentos, and Tintic have been checked by means of x -rays.

Figure 11 shows some preliminary carbonate curves. These are consistent with themselves and indicate the possible use of thermal analysis for quantitative studies of carbonate rocks. Siderite (Kerr and Kulp, 1947) and rhodochrosite yield exothermic oxidation "domes" due to the

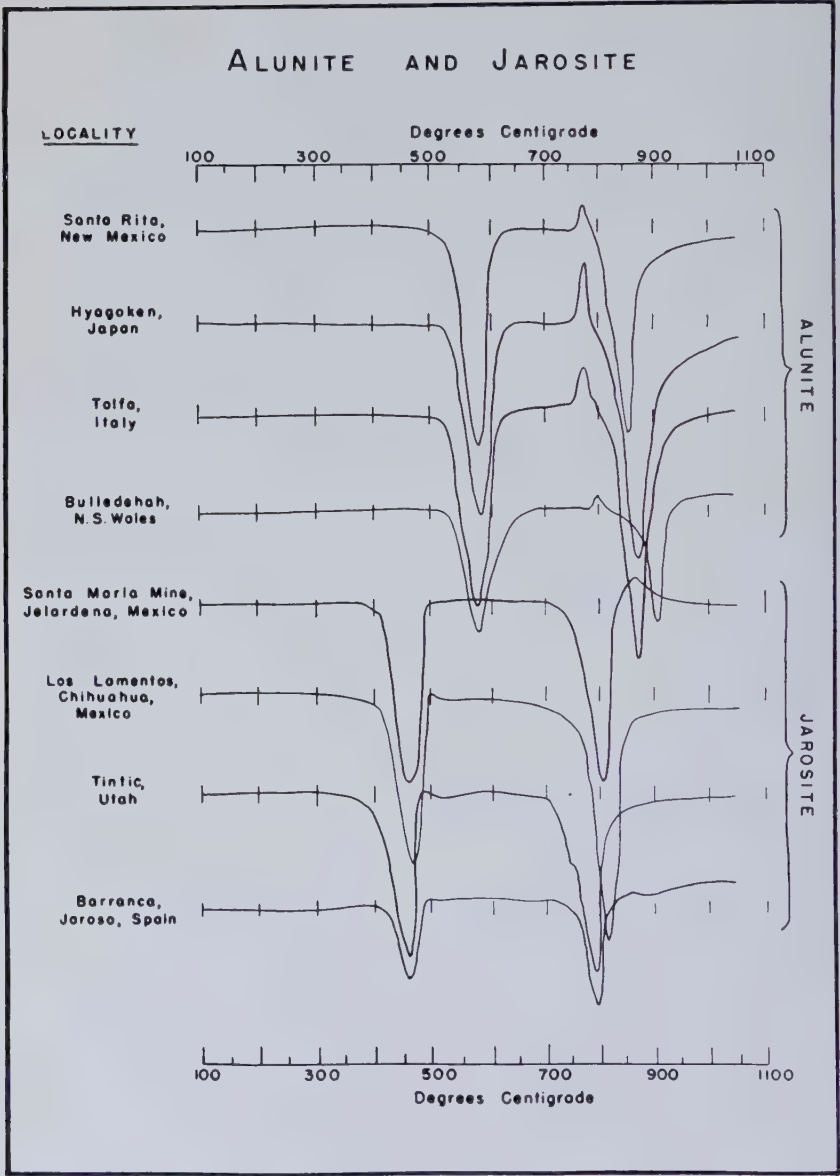


FIG. 10. Thermal curves are shown for alunite and jarosite.

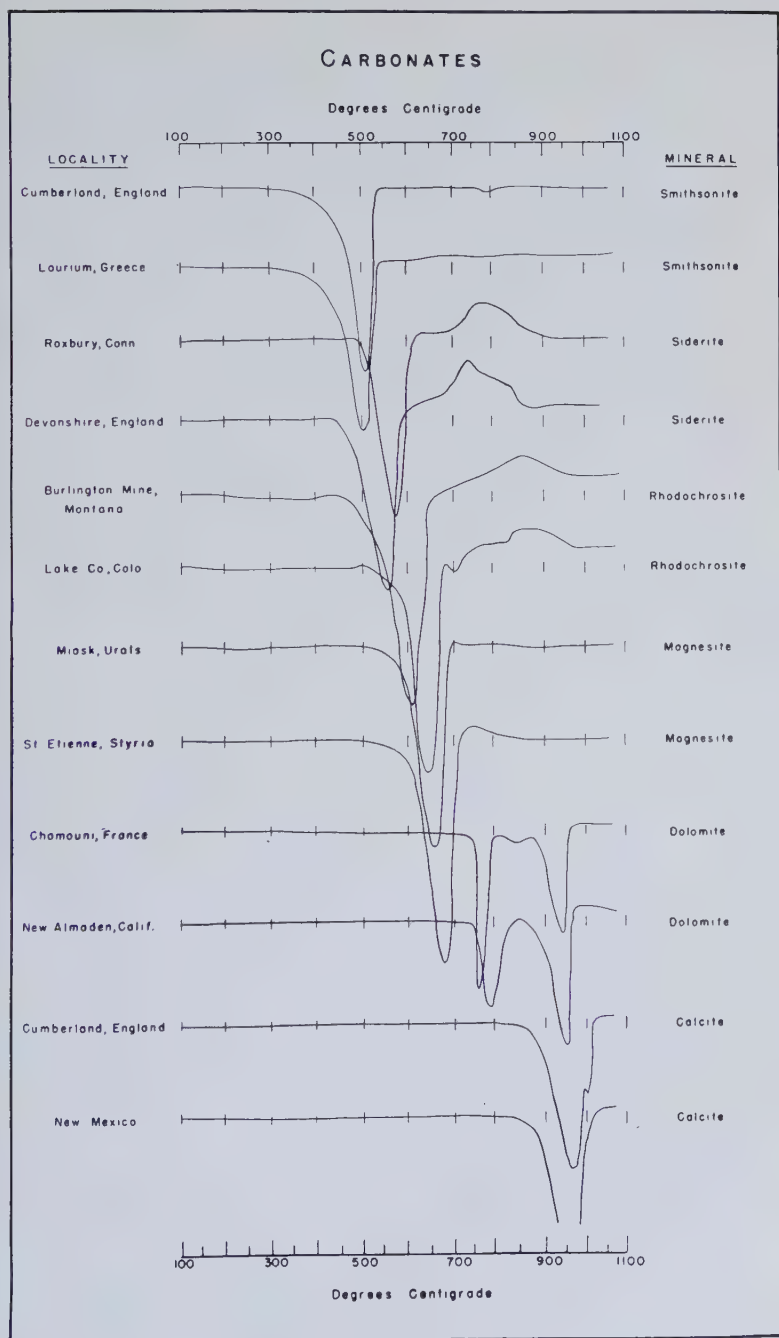


FIG. 11. Thermal curves are shown for a number of common rhombohedral carbonates.

reaction with the oxygen of the air of the lower valence oxide produced in the carbonate decomposition. Cuthbert and Rowland (1947) published thermal curves of several carbonate minerals.

In these curves the carbonate peaks are low due to the admixture of inert material. The curves from Fig. 11 closely approximate the carbonate specimens run by other workers (Speil, Berkelhamer, Pask, Davis, and Faust (unpublished)).

Artificial Mixtures

Figures 12–19 show sets of thermal curves of predetermined mixtures ground to 50 mesh.* Although theoretically the area under the curve should be proportional to the percentage of the mineral present, this does not strictly hold experimentally. It has been found, however, that for known mixtures the amplitude of the peak plotted against the percentage of the mineral present gives a smooth curve. Moreover, it has been found that this “calibration curve” is not particularly affected by the chemical nature of the other components. Using figures for artificial mixtures containing kaolinite to furnish data, the graph, Fig. 20 was prepared. The amplitude of the endothermic 605° C. peak for kaolinite is plotted against the per cent of kaolinite in the particular mixture. A different symbol is used for each mixture. The area within the two smooth curves indicates the possible error to be expected from a mixture of kaolinite with an unknown aggregation, as indicated by artificial mixtures. Clay minerals that give such distinctive peaks as kaolinite may be quantitatively estimated with reasonable certainty for simple mixtures within 10 or 20 per cent. The variation may be due in part to minor differences in the heat conductivity of the foreign constituent.

The necessary assumption to render valid the application of the calibration curve to an unknown mixture is that the clay minerals in the unknown must be in roughly the same physical and chemical condition

* The samples used in these artificial mixtures were essentially uncontaminated materials from well-known localities and were checked both optically and by means of x-ray diffraction. The alunite sample was analyzed chemically by Ledoux and Co.

<i>Mineral</i>	<i>Locality</i>
Alunite	Santa Rita, N. M.
Jarosite	Santa Maria mine, Jelardena, Durango, Mexico.
Kaolinite	Dry Branch, Georgia.
Montmorillonite	Polkville, Miss.
Quartz	—
Sericite	American Canyon, Nev.
Dickite	Cusiuhirachic, Mexico.
Goethite	Lake Superior.

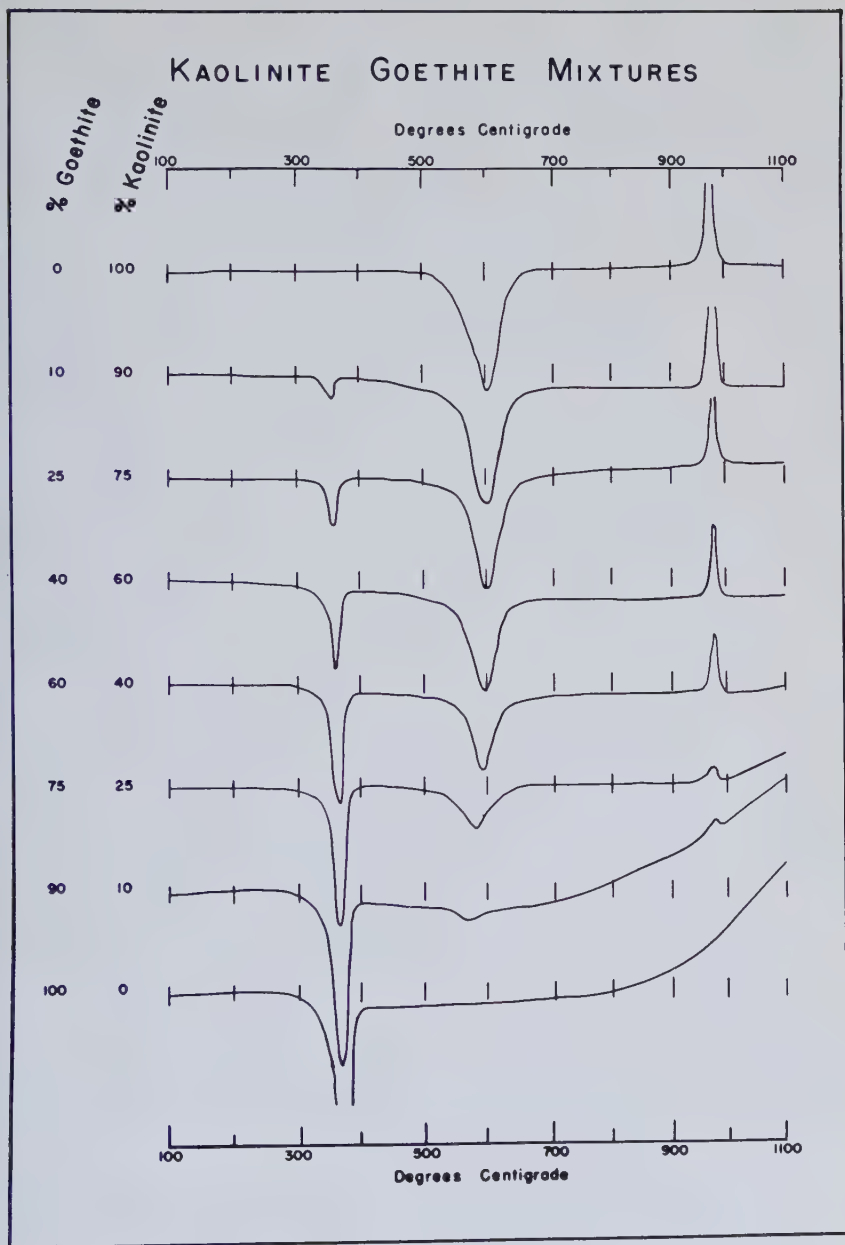


FIG. 12. Artificial mixtures of kaolinite and goethite arranged to illustrate the possible interpretation of natural mixtures.

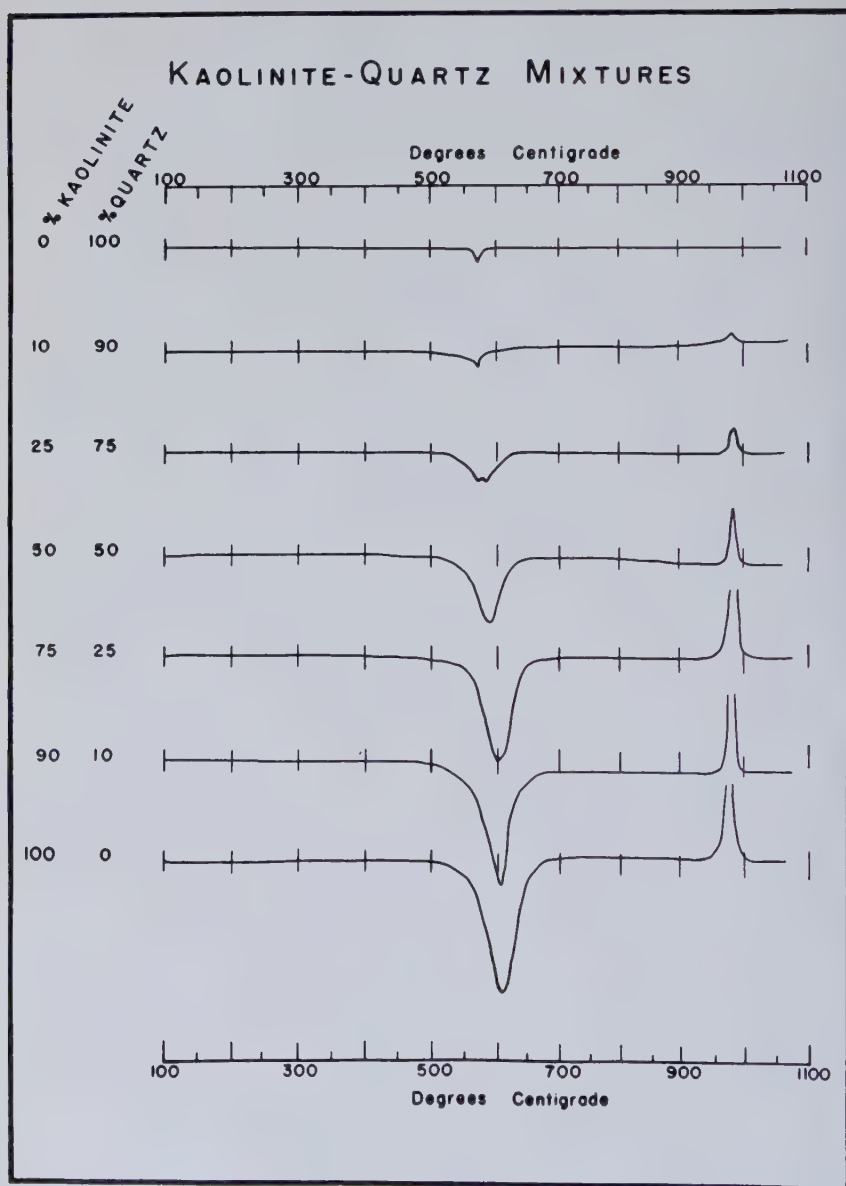


FIG. 13. Curves indicating the effect on kaolinite of quartz as an impurity.

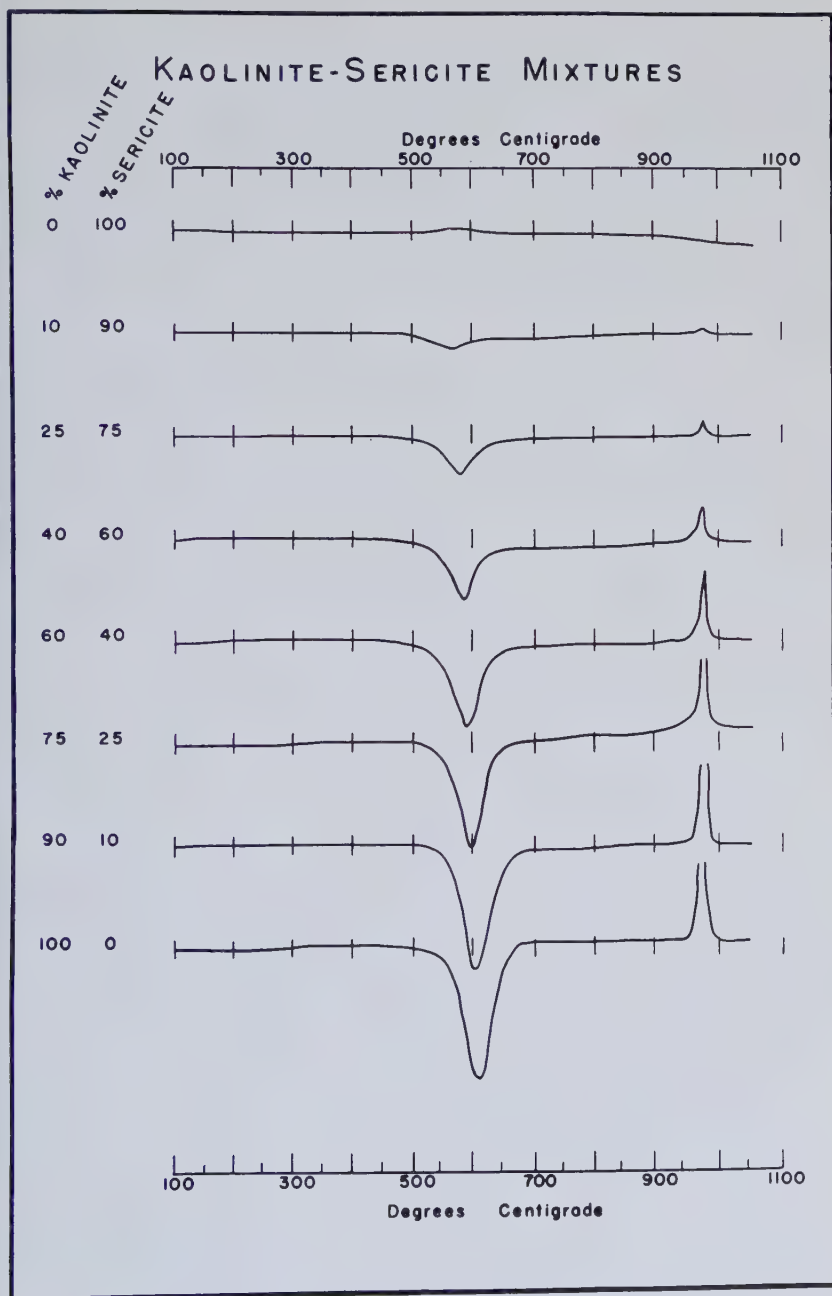


FIG. 14. Curves showing mixtures of almost inert sericite with active kaolinite.

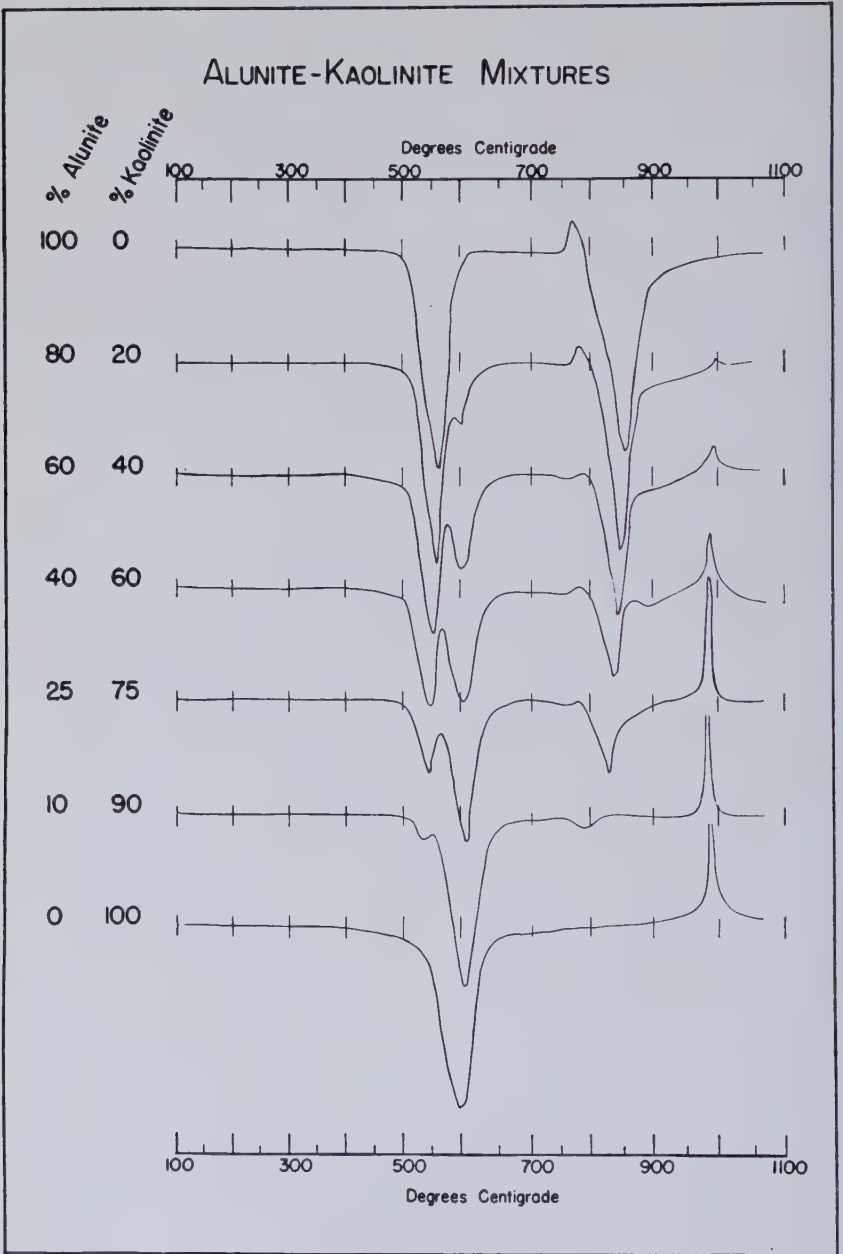


FIG. 15. Thermally active kaolinite and alunite in artificial mixtures.

ALUNITE JAROSITE MIXTURES

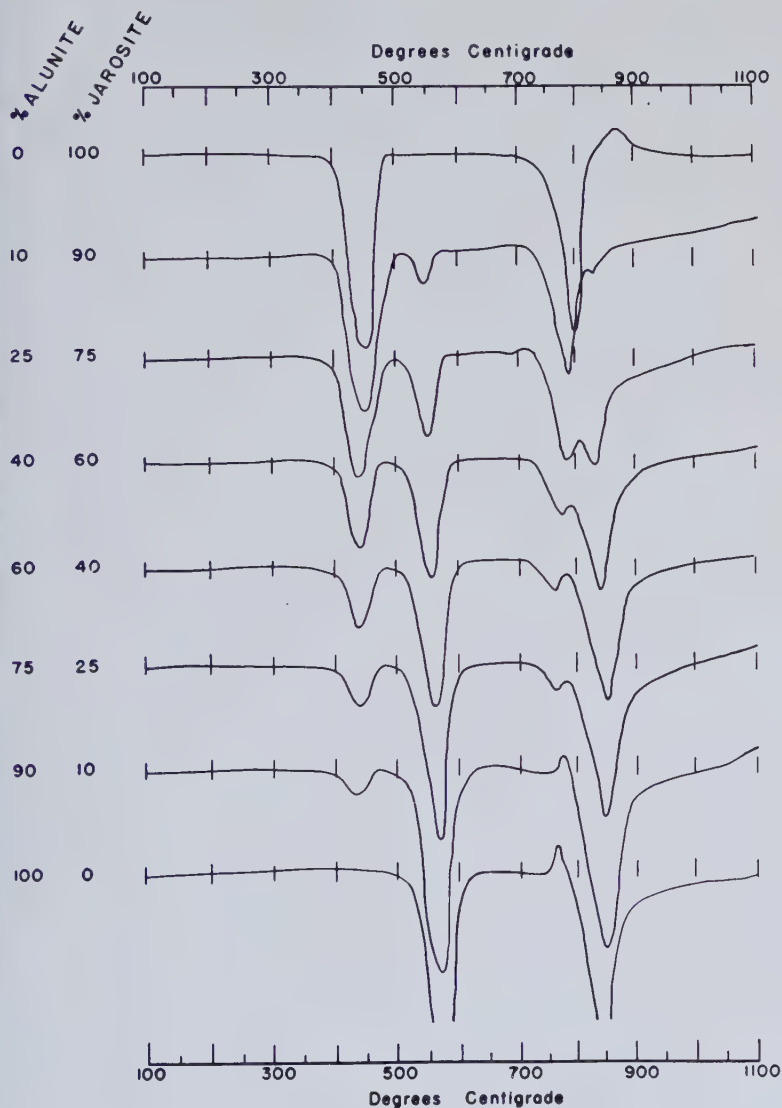


FIG. 16. Thermal curves of artificial kaolinite-jarosite mixtures.

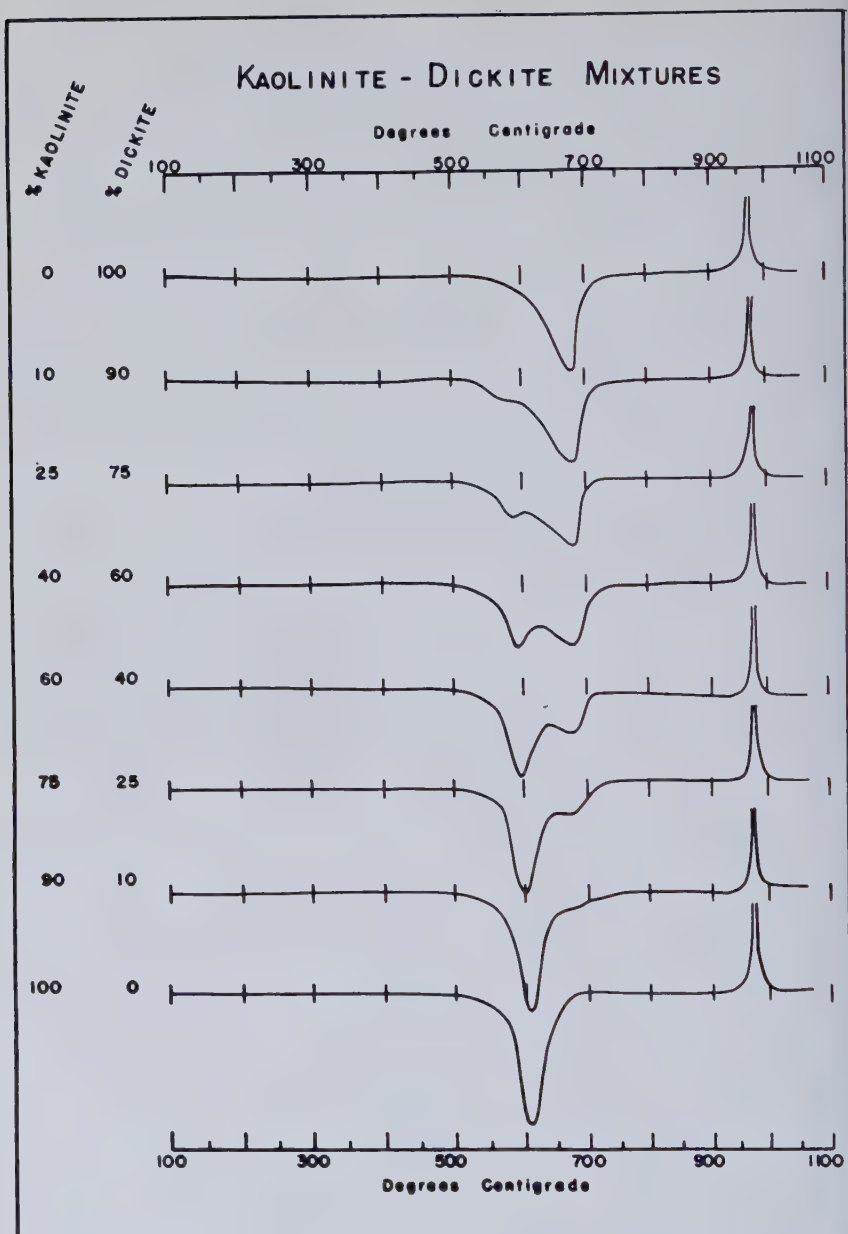


FIG. 17. Artificial mixtures of kaolinite and dickite showing the variation in thermal curves.

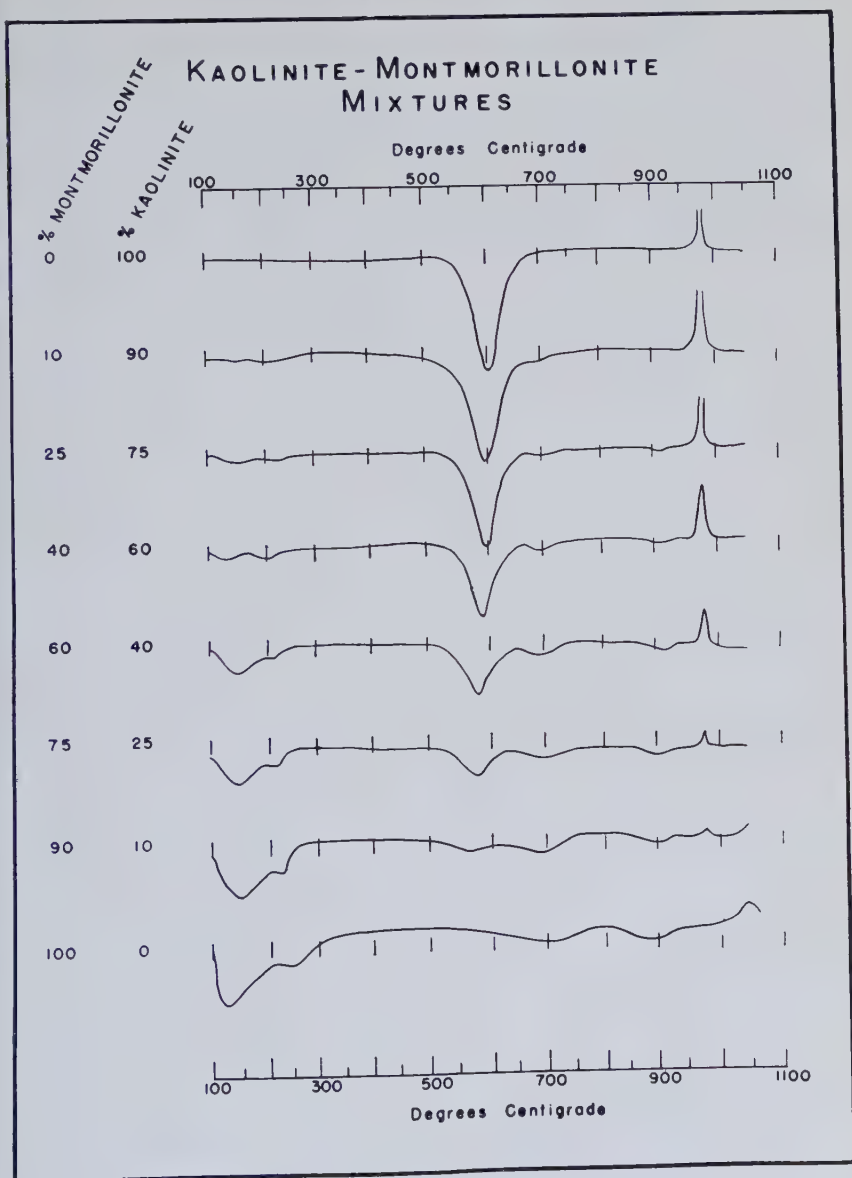


FIG. 18. Thermal curves of artificial kaolinite-montmorillonite mixtures with a range from 0 to 100 per cent.

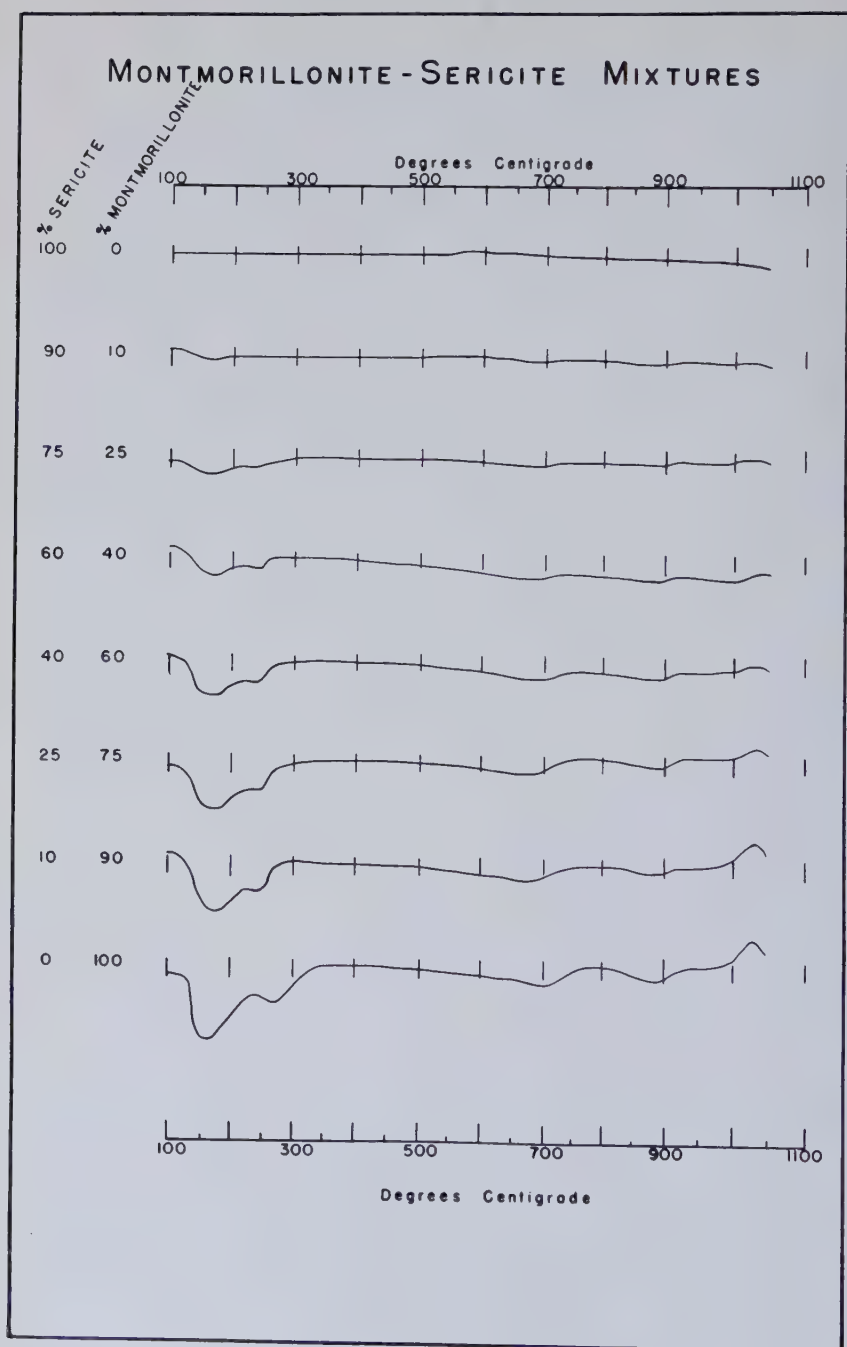


FIG. 19. Artificial mixtures of almost inert sericite with montmorillonite.

as in the artificial mixtures. This is probably a good approximation in many cases, particularly in the case of hydrothermal clays formed in situ. Grim (1947) has already pointed out the need for great caution in making such an assumption for certain sedimentary clay mixtures.

Figure 12 shows a suite of kaolinite-goethite mixtures. The endothermic decomposition peaks for both minerals are shifted down in temperature with increasing percentage of the other mineral. This shift is to be attributed to the conductance of the heat away from the particles in the endothermic reaction by the foreign inert neighbors. The 980°C . exothermic peak is not shifted appreciably. This is probably due to the narrow temperature range of the reaction. Below a certain temperature, under these conditions of molecular structure, amorphous alumina will not change over to gamma alumina. At 980°C ., however, crystallization occurs almost instantaneously. Hence, the mixture of 50 mesh inert material with 50 mesh kaolinite does not appreciably shift this peak.

Figure 13 illustrates the effect of mixing quartz with kaolinite. The quartz curve is a straight line aside from a minor peak at the inversion point. The kaolinite curve is depressed by the admixture of quartz, but comparison with Fig. 6 indicates that otherwise there does not appear to be any substantial change.

Figure 14 represents mixtures of sericite and kaolinite. Sericite shows little noticeable differential effect. On the other hand, even as little as 10 per cent kaolinite in a mixture with sericite may be detected. Since both minerals are common in zones of hydrothermal alteration, this feature is of interest.

Figure 15 contains curves of kaolinite and alunite which represent a mixture of two thermally active minerals which may occur together in the same deposits. Both minerals yield sharp and distinctive thermal peaks.

Figure 16 represents a sequence of thermal curves for alunite and jarosite where the samples are artificial mixtures. Both alunite (Fig. 10) and kaolinite (Fig. 6) are illustrated elsewhere. Where curves show such prominent peaks mixtures may be studied with reasonable facility. A proportional decrease in the amplitude as well as a downward shift of peak temperatures occurs with an increase in foreign constituents.

A common problem in the study of zones of argillic alteration concerns the estimation of the relative amounts of kaolinite and dickite present in a natural mixture. Figure 17 illustrates a series of artificial mixtures of the two minerals.

Kaolinite-montmorillonite mixtures are illustrated in Fig. 18. Evidently the apparatus as normally employed is less sensitive for the detection of montmorillonite in a mixture than it is for distinguishing minerals with higher temperature and more distinctive thermal effects.

AMPLITUDE OF KAOLINITE ENDOTHERMIC PEAK IN VARIOUS MIXTURES

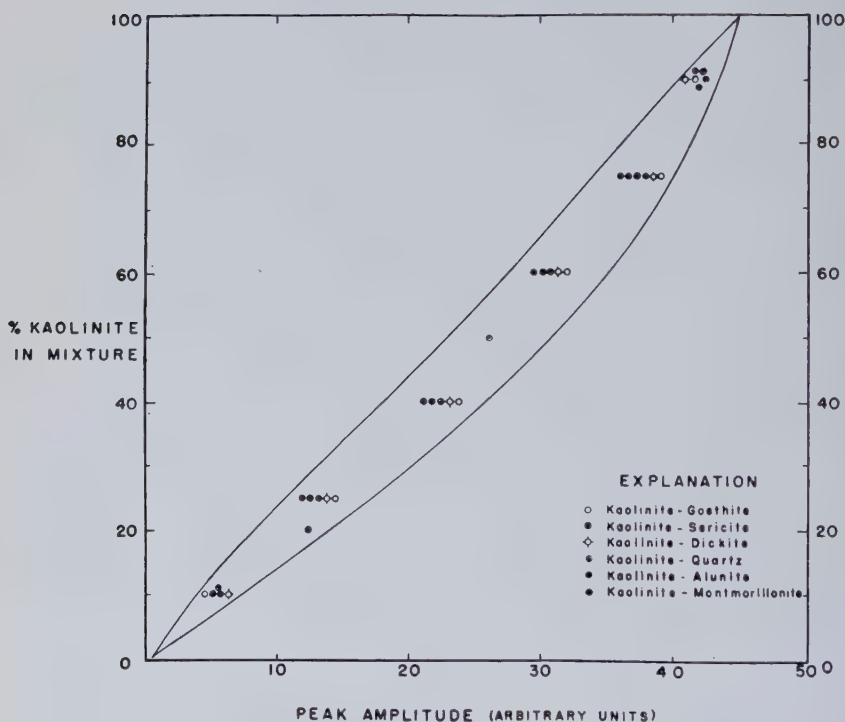


FIG. 20. A graph showing the variation in the position and amplitude of the 605° C. peak of kaolinite in various artificial mixtures.

Montmorillonite-sericite mixtures are indicated in Fig. 19. While montmorillonite would be detected in such mixtures, it seems likely that sericite would escape detection. It is evident that the effect of shifting the peaks with the per cent of impurity must be determined for each mineral to properly identify minerals in mixtures. The carbonates appear particularly sensitive to this effect.

The above curves have been used effectively in the semi-quantitative determination of the argillic constituents of an altered mineralized area. The application of the technique to this form of a problem offers significant possibilities in mapping alteration zones associated with mineral deposits in studies of the type reviewed by Kerr (1947).

The thermal curves thus far obtained in these studies are for the most part consistent with curves recorded in the literature, allowing for the

variation in heating rates. The temperatures at which peaks occur have been agreed upon by various observers with different types of apparatus, providing the heating rates, the thermal couples, and the size of sample are constant. The amplitude of the peaks for any given concentration of active ingredient is a function of the sensitivity of the individual apparatus.

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PEGMATITES OF EIGHT MILE PARK, FREMONT COUNTY, COLORADO*†

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ABSTRACT

The Eight Mile Park pegmatite district, three to twelve miles west of Canon City, Fremont County, Colorado, includes about 30 square miles that lie at the westernmost end of the Canon City embayment in the Front Range structure. The area is a dissected plateau, 6000 to 7000 feet in altitude, that is bisected by the Royal Gorge of the Arkansas River, which is nearly 1200 feet deep locally. The pre-Cambrian rocks that underlie the

* Contribution from the Harvard Department of Mineralogy and Petrography, No. 299.

† An abridged form of a thesis submitted to the Division of Geological Sciences, Harvard University, as partial fulfillment of the requirements for the degree of Doctor of Philosophy. The detailed geology of the area will be presented in a separate paper.

area are flanked on the east by hogbacks of formations of Paleozoic age and on the south and west by flat lying beds of Mesozoic rocks. Three major pre-Cambrian rock units are present: Pikes Peak granite, Idaho Springs schist, and injection gneiss.

The oldest, the Idaho Springs formation, consists chiefly of muscovite schist with minor biotite schist and quartzite. This series was intruded after metamorphism by irregular sills and lenses of hornblende gabbro. Later the batholith of Pikes Peak granite, a red, coarse-grained, porphyritic, microcline granite, was emplaced. Late differentiates from the granitic magma were aplites followed by pegmatites. The youngest pre-Cambrian rocks are dikes of diabase. Veins are not common. The injection gneiss was formed by large scale lit-par-lit intrusion of Idaho Springs biotite schists by granite, aplites, and pegmatite.

The pre-Cambrian structural elements trend uniformly northeast-southwest. They include (1) the trend of the Idaho Springs schist belt and its contacts, (2) the attitude of the schist foliation, (3) the attitude of the primary granite flow structure, (4) the strikes of aplites in granite, (5) the attitudes of pegmatite sills in schist, and (6) the trend of the axis of the major anticline in the injection gneiss.

Three types of pegmatites are present: interior, marginal, and exterior. The interior pegmatites, which are relatively small, tabular dikes within the batholith, tend to occur in swarms. They contain microcline, oligoclase, quartz, muscovite, biotite, and schorl. Not uncommonly a zonal arrangement of petrologic units is present. The marginal pegmatites, which also occur in granite but are restricted to its border, are large, flat lying, sheet-like masses or discoidal bodies that transect the granitic flow structure. Their mineralogy is similar to that of the interior pegmatites. Zonal structure is poorly developed, but locally there occur small concentrations of uncommon minerals. The marginal pegmatites occur in schist near the batholith contact. They vary greatly in size and are moderately dipping, tabular, or lens-like sills along the schistosity. Their zonal structure is well defined, and secondary units containing concentrations of sodic plagioclase, muscovite, and rarer minerals are strongly developed, especially along the footwall contacts of cores.

The zones are primary and appear to have formed by successive crystallization of pegmatite magma inward from the walls. The secondary units follow fractures or zone contacts and are believed to have formed by the replacement of preexisting pegmatite. Two hydrothermal phases can be recognized: an early sodic plagioclase-muscovite stage, and a later, more intense, but more restricted cleavelandite-lepidolite type. The total number of minerals is 35. Among the more unusual pegmatitic species are fremontite (the type locality), beyerite, triplite, and chalcocite.

INTRODUCTION

GENERAL

This study is concerned with the pegmatites and geology of Eight Mile Park, Fremont County, Colorado. It is an attempt to interrelate the structure and mineralogy of the pegmatites with the geology of the rocks in which they occur.

The writer, as a member of the U. S. Geological Survey, became familiar with the general aspects of the geology of the area through work done in late 1942 and mid-1943. A study of the detailed mineralogy of the deposits was begun in the Laboratory of Mineralogy of Harvard University in early 1946. Mapping of the individual pegmatite bodies and of the entire area was begun early in June, 1946, and was completed by

mid-September of that year. Additional laboratory work was done in the fall of 1946 at the Montana School of Mines, Butte, Montana.

The writer was visited in the field by Professors E. S. Larsen and C. S. Hurlbut, to whom he is particularly indebted for continuous assistance and careful advice from the inception of the project to its conclusion. Professor Clifford Frondel contributed many valuable suggestions and discussions. The writer is also grateful to Professor C. Wroe Wolfe of Boston University for important assistance in many aspects of the laboratory work. The cost of numerous thin sections was met by the Harvard Department of Mineralogy and Petrography, and a grant from the Brodrick Fund of Harvard University helped to defray the expenses of field work in 1946. The writer also wishes to acknowledge the generous assistance of Professor Walter F. Hunt, Editor of the *American Mineralogist*, in converting the study from thesis form to one suitable for publication, and that of Edith Dunn Heinrich in all aspects of the manuscript preparation. Mr. M. V. Denny kindly made several important photographs.

PREVIOUS WORK

The earliest record of mica deposits in Fremont County, Colorado, was made by Albert Williams (1883),¹ but no specific deposits are mentioned. Headden (1905) described columbite from the Eight Mile Park pegmatites, and in 1911 Schaller (1911, 1912, 1914, 1916) presented the first of his descriptions of fremontite, a mineral that has been found in only one other locality. Sterrett (1913, 1923) examined many Colorado pegmatites shortly before World War I. No further work was done until 1932 and 1933, at which time Landes (1935, 1939) examined several pegmatite districts in Colorado, including the Eight Mile Park area.

The general geology has been described by Darton (1906) and also by Campbell (1922). Powers (1935) studied the physiography of the Royal Gorge, and in 1941 brief notes on the geology were made by Kessler (1941). Blum (1944, 1945, 1946) has made a magnetic survey of the area. Special studies involving the sedimentary rocks have been completed by Walcott (1891), Washburne (1908), and Tieje (1923).

GEOGRAPHY

LOCATION AND ACCESS

The Eight Mile Park area is in Fremont County, Colorado, 3 to 12 miles west and northwest of Canon City (Fig. 1). The area mapped, which consists of about 30 square miles, extends from 105° 15' to 105° 22' 30"

¹ References are at the end.

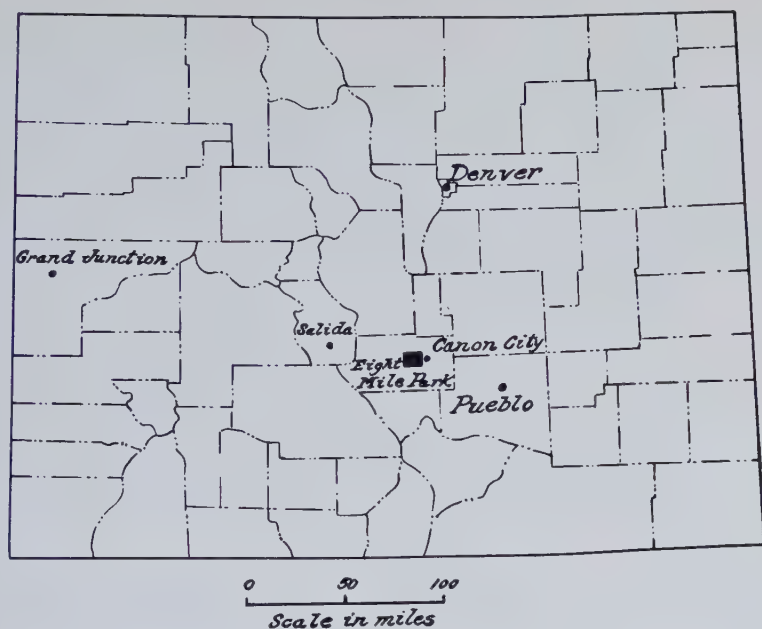


FIG. 1. Index map of Colorado, showing location of Eight Mile Park pegmatite district.

West Longitude and from $38^{\circ} 26'$ to $38^{\circ} 30'$ North Latitude. It lies on both sides of the Royal Gorge of the Arkansas River.

The region is easily accessible over U. S. Highway 50, which parallels the eastern and northern sides. From it the Royal Gorge road extends southward to the Royal Gorge bridge and beyond to the Webster Park road. State Highway 9 and the Webster Park road skirt the western and southern margins. The southern line of the Denver and Rio Grande Western Railroad from Denver to Salt Lake City passes through the Royal Gorge.

PHYSICAL FEATURES

Eight Mile Park is a plateau of resistant rocks bounded except on the north by softer sedimentary formations, which dip away in a quaquaversal manner. The southeastward flowing Arkansas River has cut a deep and narrow gorge through the center of the area. Elevations range from about 7000 feet at the top of Fremont Peak to 5350 feet at river level at the east end of the Gorge.

The highest and most rugged part of the plateau is in the southeastern corner. Westward and northwestward it changes gradually into moderately and gently rolling country with extensive park-like areas that be-

gin at an elevation of about 6100 feet and slope gently westward where they pass into the sharply dissected margin along the sedimentary contacts. Powers (1935, p. 190) states that "The plateau retains parts of two erosion surfaces—the eastern upland about 6800 in altitude and the 6100-foot 'flat area'." However, the two sections are not distinctly separated, and the former is too poorly defined to be easily recognized as the remnant of a separate erosion surface. Probably there is represented only a single, westward dipping erosion surface whose higher eastern part has been more strongly dissected.

PETROLOGY

GENERAL STATEMENT

The pre-Cambrian rocks may be grouped into three major units which occur as northeast-trending belts. From northwest to southeast these are:

1. Pikes Peak granite
2. Idaho Springs formation
3. Injection gneiss

The oldest rock is the Idaho Springs formation which was intruded by Pikes Peak granite. Large-scale lit-par-lit injection by granitic material related to the Pikes Peak magma fashioned the injection gneiss from selected parts of the Idaho Springs formation. Closely related to the intrusion of Pikes Peak granite are dikes of aplite, aplitic granite, pegmatite, and diabase. The pegmatites are described in detail in a special section.

IDAHO SPRINGS FORMATION

The name Idaho Springs formation was applied by Ball (Spurr, Garrey, and Ball, 1908) to a highly metamorphosed series of rocks that forms the oldest unit of the pre-Cambrian complex in the Georgetown Quadrangle. Four lithologic units were distinguished: biotite-sillimanite schist, biotite schist, quartz gneiss (impure quartzite), and lime-silicate rocks. Similar rocks had been described previously by Cross (1894A, 1894B) from the Pikes Peak and Crested Butte Quadrangles. Finlay (1916) indicated the presence of small areas of these rocks in the Cripple Creek Quadrangle but did not differentiate them on his map. Later Loughlin and Koschmann (1935) described them in detail. Lovering (1929) correlated the earlier descriptions in his summary of the geologic history of the Front Range.

In the Eight Mile Park area the Idaho Springs formation is chiefly a muscovite schist with variable but generally minor amounts of biotite schist, biotite-muscovite schist, biotite-sillimanite schist, quartz-mica schist, quartzite, and exceedingly rare layers of quartz-epidote rocks.

The most typical phase is a coarse-grained muscovite-rich schist in

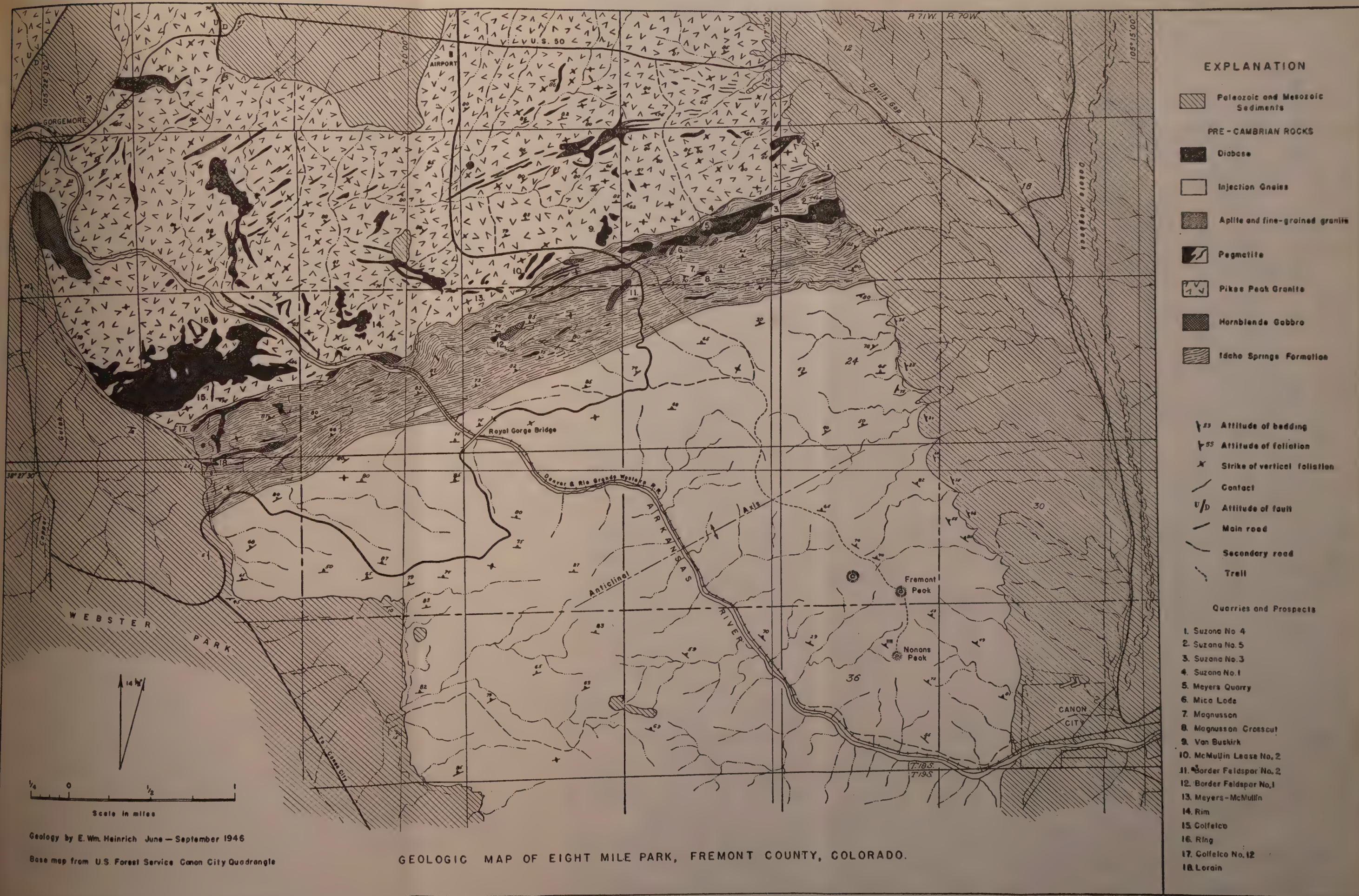


FIG. 2

which the mica flakes may attain a diameter of $\frac{3}{8}$ -inch. A similar fine-grained rock is comparatively rare. The color ranges from silver to blue-gray to grayish-green, and the schistosity is commonly well developed. Quartz is the only other common mineral, feldspar occurs sparingly, and biotite may be present in variable quantities. Under the microscope this rock is seen to consist chiefly of muscovite and quartz. Iron-stained sericite is very abundant and replaces both quartz and earlier coarser muscovite. Accessory minerals include abundant magnetite which is commonly fractured and veined by sericite, a little chlorite, traces of sillimanite, and a few grains of zircon. Zircon was noted in quartz-biotite schists in the Cripple Creek Quadrangle (Finlay, 1916, p. 4). Locally this rock with an increase in the amount of quartz grades into quartzite. Schists that contain important quantities of both muscovite and biotite are rare.

The Idaho Springs formation has been intruded by sills of gabbro with the production of quartz-hornblende contact rocks containing plagioclase metacrysts. Other contact schists contain coarse metacrysts of muscovite or $\frac{1}{8}$ -inch muscovite "knots" in a fine-grained groundmass of biotite and quartz.

Near bodies of aplite epidote may be abundant, and tourmaline metacrysts have been formed along the margins of some small pegmatite bodies. Ball (Spurr, Garrey, and Ball, 1908, p. 40) states that "... large prisms of black tourmaline are embedded at right angles to the schistosity near pegmatite dikes containing similar tourmaline." The larger pegmatities commonly are sheathed in recrystallized and coarsened muscovite schist.

HORNBLLENDE GABBRO

Sills and lenses of hornblende gabbro, which range in length from 400 to 2500 feet and in width from 20 to 350 feet, have been intruded into the Idaho Springs formation. The largest body occurs north of the mouth of Copper Gulch, where it apparently forms part of a roof pendant in Pikes Peak granite. This mass, which is 3300 feet long and 1000 feet wide, has been intruded and altered by the granite. Thus the gabbro is younger than the Idaho Springs formation and older than the granite. The foliation is very poorly developed, and the sills were intruded toward the close of or after the metamorphism of the Idaho Springs schists. Mafic hornblende-bearing rocks in the injection gneiss probably represent altered remnants of similar gabbroic sills.

The most common rock type is a dark, coarse-grained hornblende gabbro that contains varying amounts of biotite. The grain sizes ranges from $\frac{1}{8}$ -inch to $\frac{5}{8}$ -inch, but the coarser phases are of local extent only. In addition to this predominant type there occur:

1. Quartz-hornblende gabbro that resembles the normal type except for the presence of quartz. This rock appears to have been formed by reaction with quartz-rich schist, for it is a marginal phase of some sills.
2. Hornblende in which plagioclase is a very minor constituent.
3. Biotite-bearing hornblende in which plagioclase is likewise subordinate.
4. A biotite-rich rock which contains abundant magnetite and apatite. Plagioclase is subordinate and hornblende is absent.
5. A rare biotite-rich rock which contains magnetite, olivine, and traces of hornblende and plagioclase. The grain size averages $\frac{1}{4}$ inch.
6. Local highly schistose phases, which are probably the result of post intrusive fracturing and shearing, contain abundant chlorite and some secondary quartz. Some are marginal to large bodies of pegmatite.

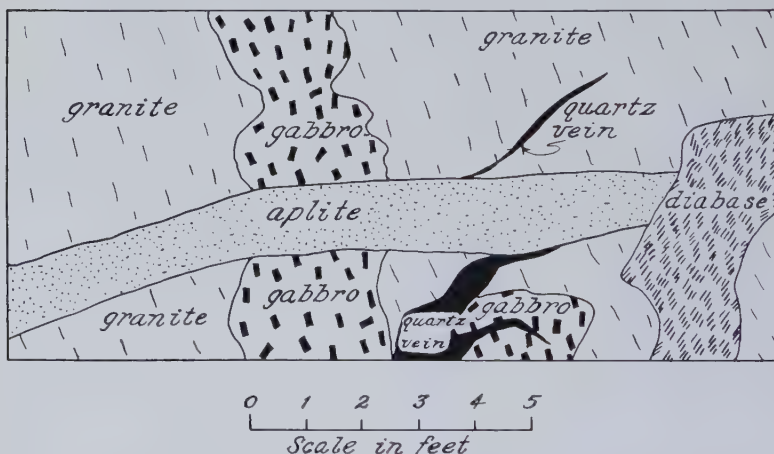


FIG. 3. Age relationships between gabbro, Pikes Peaks granite, aplite, diabase, and quartz veins, Copper Gulch.

Microscopically the gabbro consists of an equigranular aggregate of hornblende, labradorite, and minor biotite. Magnetite and apatite are common accessories. Chlorite and calcite are products of weathering. Some of the hornblendes are poikilitic with plagioclase inclusions. The plagioclase ranges in composition from calcic andesine to calcic labradorite.

Associated with the gabbro are two minor types of pegmatite. The first, which was observed just west of the Border Feldspar No. 1 pegmatite, contains abundant quartz and chlorite and minor microcline. The brownish-green chlorite appears to be primary. The other variety occurs in one- to two-foot pods marginal to the thick sill of gabbro in the gulch northeast of the Lorain quarry in the SW. $\frac{1}{4}$, SE. $\frac{1}{4}$, Sec. 20. It consists of abundant hornblende in two-inch blades, epidote, albite, apatite, and traces of biotite.

The gabbro has been intruded by Pikes Peak granite, aplite, pegmatite, and diabase (Figs. 3 and 4). Around the margins of aplite dikes it contains abundant epidote, calcite, and red microcline in veinlets and minute blebs.



FIG. 4. Xenoliths of gabbro in Pikes Peak granite, Copper Gulch.

PIKES PEAK GRANITE

The most widespread igneous rock is the Pikes Peak granite, which underlies the northern third of the area. The Pikes Peak granite was first described by Mathews (1894) and then shortly thereafter by Cross (1894A). It was subsequently found to have a wide distribution in the Colorado Front Range, especially in the south-central part where it forms a batholith about 80 miles long and nearly 35 miles wide. Lovering (1929) has correlated the Pikes Peak with the Rosalie granite, and Boos (1934) has indicated that it is also probably identical with the Sherman granite in northern Colorado and Wyoming.

The Canon City body is separated from the main batholith by an arm of Cripple Creek granite on the north and by the down-faulted block of sediments in Garden Park on the east.

The main rock type is a red, coarse-grained, porphyritic granite, which forms the central part of the mass. The groundmass, whose average grain size is about $\frac{3}{16}$ -inch, consists of quartz, oligoclase, and abundant biotite. In this matrix are set well formed phenocrysts of red microcline,

which range in length from $\frac{1}{2}$ inch to 2 inches, with an average of about 1 inch. Rarely are they less than $\frac{1}{2}$ inch long. Accessory minerals are abundant allanite and sphene, apatite, and magnetite. Fluorite, which is a common accessory mineral of this granite in the vicinity of Pikes Peak (Mathews, 1900), appears to be absent in the Eight Mile Park area. Biotite is commonly partly altered to chlorite, and the feldspars, especially the plagioclase, may be thoroughly sericitized. A small amount of micropegmatite is present.

Table 1 presents Rosiwal analyses of four specimens of Pikes Peak granite.

TABLE 1. ROSIWAL ANALYSES OF PIKES PEAK GRANITES

	A.	B.	C.	D.
Microcline and orthoclase	52%	42%	56%	66%
Quartz	23	29	33	22
Oligoclase	13	8	4	2
Biotite	10	12	4	7
Muscovite	tr	6	tr	tr
Accessory minerals	2	3	3	3
	100	100	100	100

A. Royal Gorge, Mouth of Overshot Gulch, Eight Mile Park, Fremont County, Colorado.

B. U. S. Highway 50, 3 miles west of Parkdale, Fremont County, Colorado.

C. Idem.

D. East side of Litterdale Gulch, near Lake George, Park County, Colorado.

The normal granite has a well-defined foliation characterized by a planar orientation of the tabular phenocrysts and platy biotite. Along the schist contacts a finer-grained, non-porphyritic, gneissic phase is prominently developed. Biotite is more abundant. Local porphyritic patches occur, and the contact with the normal phase is gradational.

The Pikes Peak granite was intruded successively by:

1. Sills, dikes and small stocks of aplite and fine-grained granite.
2. Dikes of pegmatite.
3. Dikes of diabase.

The magmatic activity continued with the formation of several types of veins:

1. Rare allanite veinlets as much as $\frac{3}{4}$ inch thick.
2. Veinlets of flesh colored feldspar with minor quartz. Younger epidote, which may be coated by fine-grained blue-green chlorite, is also present in many.
3. Veinlets of epidote.

4. Veinlets and lenses of quartz. Large pods of "bull" quartz are restricted to the schist-granite contacts. The quartz veinlets contain plagioclase, black tourmaline, hornblende, chlorite, biotite, and ilmenite as rare constituents.

The formation of these veins was followed by a period of fracturing during which well-defined narrow shear zones were developed in a few places. Elsewhere parts of the granite body were sheared in a wholesale manner with the development of cataclastic structures and minor mineralization. A few unmineralized fractures with slickensides were also noted, but these may be much younger.

APLITE AND FINE-GRAINED GRANITE

Sills and dikes of aplite and fine-grained granite are the oldest of the minor differentiates that intrude the Pikes Peak granite. Sills along the

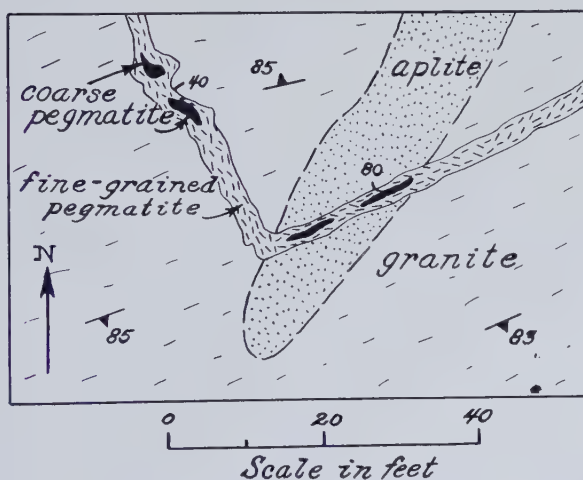


FIG. 5. Pegmatite transecting aplite, Overshot Gulch.

granitic foliation are more common than dikes and these form the larger masses. Some of the smaller aplite dikes were intruded along the contacts of tabular schist xenoliths in granite. A few small stocks, some with concordant apophyses, were also mapped. In addition, aplite sills and dikes have been intruded into the schists of the Idaho Springs formation and into the hornblende gabbro. Several large pod-like bodies of aplite have been localized along the Pikes Peak-Idaho Springs contact. Several generations of aplite are present. The earlier bodies tend to be sills along the granitic foliation, whereas the later ones were injected along fractures.

The most important rock type is a fine-grained aggregate of red micro-

cline and gray quartz. Biotite and plagioclase are very subordinate or absent. A little muscovite may be present, replacing biotite with the formation of magnetite "dust." This type grades into a fine-grained equigranular granite that contains, in addition to abundant quartz and microcline, the essential minerals, plagioclase, orthoclase, and biotite. Muscovite, apatite, zircon, and magnetite are common accessories.

Most of the aplites and aplitic granites are red in color, owing to the predominance of red microcline and orthoclase. Some of these rocks, in which biotite is more abundant, are light gray in color.

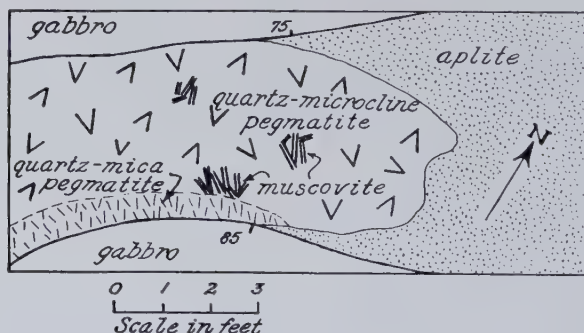


FIG. 6. Composite aplite-pegmatite sill, near Meyers Quarry.

Although the aplites are generally somewhat earlier than the pegmatites, which transect them (Fig. 5), gradations from aplite to pegmatite were noted (Fig. 6). A few aplite dikes contain central pods of massive white quartz, similar in appearance and arrangement to the massive quartz cores of many of the pegmatite bodies.

Some aplitic sills in granite developed strong gneissic structures parallel to their strike and dip. The structures appear to be primary, for the surrounding granite is undeformed as are later diabase dikes which transect the aplite sills.

DIABASE

Dikes of diabase occur throughout the entire area of preCambrian rocks. Several textural varieties are present. The smaller bodies consist of a dense, brown to gray aphanitic type, which may contain conspicuous small needles of plagioclase. The larger dikes are characterized by the typical ophitic texture in which the individual grains may attain a length of $\frac{1}{4}$ inch. The aphanitic type is highly fractured and disintegrates to blocky, angular fragments, reddish gray in color, but the coarse-grained varieties are massive and weather to large spheroidal boulders which are enclosed in thin shells of decomposed, iron-stained material. In some dikes both phases are present.

Diabase dikes are especially abundant in granite but also are numerous in schist and in the injection gneiss complex. In the gneiss they were not mapped individually. The dikes are generally flat-lying, thin tabular bodies, which cut across the primary granite foliation at nearly right angles. Dips are low, ranging from 0° to 25° . One flat-lying dike, which ranged in thickness from one to about ten feet, was traced for over one quarter mile. Some dikes split into one or more arms, each of which may divide into numerous thin stringers. The largest bodies, which contain the coarse-grained phases, are most irregular in shape. Many are flat lying to moderately dipping dikes that vary widely in thickness and attitude over short distances.

A third structural type is represented by steeply dipping dikes rather uniform in attitude. These are generally 5 feet or less in width and several score feet to several hundred feet long. One set strikes N. 10° W. to N. 20° E. and the other strikes nearly east-west. Evidence of some displacement along one of the north-south fractures is found at the West Dell prospect, where an east-west pegmatite in granite has been cut off by a north-trending dike of diabase. The relationship between the steeply dipping and the flat lying dikes is well shown in the main tributary to Catlin Gulch (SE. $\frac{1}{4}$, SE. $\frac{1}{4}$, sec. 20), where a nearly vertical apophyse extends upward from a horizontal body. The vertical sill has weathered away to form a "chimney" along the schistosity.

INJECTION GNEISS

The most widespread rock is injection gneiss, which underlies the southeastern half of the area. Biotite schists of the Idaho Springs formation first were intruded by sills of gabbro and later, chiefly in lit-par-lit manner, by exceedingly numerous bodies of aplite, fine-grained granite, and pegmatite. In many places the alternation of granitic material with granitized schist is so marked that the composite appears stratified. Numerous dikes of diabase cut across this injection structure. In some horizons the structure is crumpled and very complex, with ptigmatic folds that range in size from a few inches to scores of feet. Here cross-cutting masses of granitic material are more common. A few post-diabase faults were also noted.

The pegmatitic material in the injection gneiss differs from that elsewhere in the area. It occurs in very irregular masses that consist chiefly of microcline and quartz. Small flakes of muscovite may be present, and biotite is abundant locally in plates one inch or less in width. Magnetite is unusually abundant, commonly in well-shaped octahedra as large as one inch. Garnet occurs sparingly. Patches of chlorite and veinlets of specular hematite were also observed. Graphic granite appears to be ab-

sent. Associated with the pegmatites are veins of quartz, some of which contain abundant large magnetite crystals; veinlets of quartz and hornblende; and veinlets of quartz, hornblende, and biotite.

The host rock for this large scale injection appears to have been chiefly fine-grained biotite schist with scattered lenses of hornblende gabbro. The biotite rock has been intensively granitized throughout much of the complex, resulting in the formation either of biotite schist or gneiss rich in quartz and feldspar, or of banded biotite-feldspar gneisses. The gabbroic rocks have been converted to biotite-hornblende gneisses which contain varying amounts of magnetite, quartz, and feldspar, but in general they have been altered less than the schists.

The granitic material that constitutes the major part of the injection gneiss is believed to have been derived from the Pikes Peak magma. In appearance, occurrence, and mineralogy the rocks resemble closely the late granitic rocks as they occur in the other rock units of the area—the Pikes Peak granite, the Idaho Springs formation, and the hornblende gneiss. The difference lies largely in the intensity of the injection process and in the amount of material introduced. It seems rather unusual that the most intense injection occurred at some distance from the main body of Pikes Peak granite and thus left an undisturbed belt of schists adjacent to the contacts of the batholith. Yet similar sequences of injection gneiss, schist, and Pikes Peak granite are to be observed in two places along U. S. Highway 50 between Parkdale and Cotopaxi, west of the area. It is possible that further light may be shed on this peculiar distribution when detailed mapping is continued to the south of the area.

PALEOZOIC AND MESOZOIC SEDIMENTS

The area is bounded on the east by hogbacks of Paleozoic sediments that dip gently to moderately eastward. On the west, northwest, and southwest Mesozoic beds, which begin with the Morrison formation, lie along the margins (Fig. 2). Contacts between the pre-Cambrian and the younger rocks usually are erosional in nature. Small islands of capping Morrison strata occur at several widely scattered places (Fig. 2). Faulted contacts are rare. A few small faults cut sediments and granite in the northwest corner of the area (Fig. 2) where the Mesozoic strata are locally overturned.

STRUCTURE

PRE-CAMBRIAN STRUCTURE

The pre-Cambrian rock units are characterized by a uniform structure that strikes N. 50–80° E. and is steeply dipping to vertical. The elements that combine to produce this structural uniformity are:

Idaho Springs Formation

1. Attitude of schistosity.
2. Strike and dip of pegmatite and aplite sills.
3. Attitude of fractures in pegmatites.
4. General trend of entire schist belt and of contacts with flanking rock units.

Hornblende Gabbro

1. General trend of the gabbro sills.
2. Attitude of poorly developed foliation.
3. Strike and dip of pegmatite and aplite bodies.

Pikes Peak Granite

1. Attitude of primary foliation.
2. Attitude of aplite sills.
3. Attitude of primary foliation in the sills of aplitic granite.
4. Strike and dip of fractures in aplite sills.

Injection Gneiss

1. Axis of major anticline.
2. Strike and dip of foliation on northwest flank of anticline.

LARAMIDE STRUCTURE

The Eight Mile Park area lies at the extreme western end of the Canon Embayment in the Front Range structure. No important periods of diastrophism occurred in Paleozoic time. During the Laramide orogeny the Eight Mile Park plateau was uplifted as part of the Front Range accompanied by arching of the sedimentary cover. Faulting was confined to the northwest corner of the area, where several small blocks of Mesozoic formations were dropped down relative to the pre-Cambrian rocks.

GEOLOGIC HISTORY

The schists of the Idaho Springs formation are believed to be sedimentary in origin and probably were formed by the erosion of granitic rocks no longer exposed (Lovering, 1929). Very likely the formation consisted of marine shales and sandstones; very little limestone was deposited in the Eight Mile Park area. If no isoclinal folding is present, the thickness may be as much as 10,000 feet. Lovering (1929) states that it may attain a thickness of 25,000 feet.

Uplift and strong folding followed deposition, and the sediments were transformed into schists and quartzites with a foliation parallel with the original bedding. The sills of gabbro were intruded toward the close of the period of metamorphism.

The emplacement of the Pikes Peak batholith followed, and satellitic dikes and sills of aplite and aplitic granite were injected close to the end of the period of granite crystallization. The pegmatite bodies that followed were intruded along cooling fractures in the granite as dikes (interior and marginal pegmatites), and as sills (exterior pegmatites) along the schistosity of the bordering Idaho Springs formation. The

diabase dikes followed fractures both in granite and the metamorphic rocks. During the hydrothermal stage, which was of feeble development, a variety of quartz-rich veins were formed.

Uplift and a long period of erosion followed. The erosion surface may have been largely developed at this time. Minor deposition of Paleozoic sediments began in upper Cambrian time. In the area west of Canon City the Cambrian is largely absent and probably was eroded away before deposition of Ordovician sediments was initiated. The Ordovician overlaps the Cambrian remnants westward. No Silurian or Devonian formations occur in the Canon embayment, and the Mississippian rocks (Millsap limestone) appear to be bounded both at the top and bottom by erosional unconformities. The Fountain formation of Pennsylvanian age probably did not extend far west of Canon City nor did the Permian, Triassic, or Jurassic. The Cretaceous formations overlap far to the west resting on pre-Cambrian rocks on the west side of Eight Mile Park.

During the Laramide orogeny the Eight Mile Park area was uplifted with maximum elevation near the southeastern corner. The Arkansas River began to cut its canyon and stripping of the sedimentary cover was begun.

Table 2 compares the pre-Cambrian geologic history of Eight Mile Park area with other areas in the Front Range.

PEGMATITES

DISTRIBUTION

Pegmatites occur in all three major rock units: granite, schist, and injection gneiss. The pegmatitic material in the gneiss is characterized by irregular patchy distribution and cannot be represented as distinct map units. It is of no commercial importance and of little mineralogical interest. The other pegmatite bodies in the area can be classified readily into three types: interior, marginal, and exterior. This grouping is analogous to that proposed by Gevers (1936) for the Namaqualand pegmatites. Before comparing the characteristics of each of the groups it is necessary to discuss in some detail the general internal structure of pegmatite bodies.

A magnetic survey of the Canon City Quadrangle has been completed by Blum (1944) who states (p. 557), "The second feature on the magnetic map which is of interest is the set of magnetic highs in the northwest quadrant of the map. The two western highs correlate quite well with the pegmatitic dikes which are here amply exposed. The western high in the group is situated in the sedimentary horizon and probably reflects the existence of these same dikes under the Paleozoic rocks."

TABLE 2. SUMMARY OF PRE-CAMBRIAN GEOLOGY OF SELECTED AREAS
IN THE FRONT RANGE OF COLORADO

A. Front Range (general)	B. Longs Peak- St. Vrain	C. Indian Creek	D. Cripple Creek	E. Eight Mile Park
	Mount Olym- pus granite			
Silver Plume granite (gripple Creek, Longs Peak granites)	Longs Peak granite	Injection gneiss Indian Creek granite	Cripple Creek granite	
Injection gneiss				Injection gneiss
Pikes Peak granite (Rosalie, Sherman granites)		Pikes Peak granite	Pikes Peak granite	Pikes Peak granite
Metamorphism	Metamorphism	Metamorphism		
Granite gneiss				
Quartz-Monzonite gneiss	Boulder Creek granite gneiss	Mount Evans quartz monzonite	Olivine Syenite?	Hornblende gabbro
Metamorphism	Metamorphism	Metamorphism	Metamorphism	Metamorphism
Idaho Springs fm.	Idaho Springs fm.	Idaho Springs fm.	Idaho Springs fm.	Idaho Springs fm.

A. Lovering (1929).

B. Boos and Boos (1934).

C. Boos and Aberdeen (1940).

D. Loughlin and Koschmann (1935).

E. Heinrich (this paper).

With this hypothesis the writer cannot agree. The largest pegmatite bodies flank the Pikes Peak-Idaho Springs contact to form a belt that trends east-northwest. Probably this belt continues eastward beneath the sedimentary cover. The western magnetic high referred to by Blum does not overlie the pegmatite belt nor does it reflect its shape. In all likelihood, therefore, its existence must be ascribed to other factors as yet undeciphered. For similar reasons the eastern high cannot be laid to the influence of pegmatites beneath the sedimentary cover.

GENERAL STRUCTURAL FEATURES OF PEGMATITES

History

The early history of pegmatite investigation was marked by intensive mineralogical and crystallographic study, wherein suites of specimens were collected in a generally haphazard fashion and subjected to detailed scrutiny in the laboratory. Many of the specimens were selected because of their unusual mineralogy, and in many cases, less spectacular material that constituted the bulk of the pegmatites was ignored. The results of such work were sometimes embodied in a paragenetic history, which, although complete in itself, failed to relate the mineralogy of the rarer constituents to the structure of the pegmatite or to its geologic environment. Thus, "specimen" mineralogy and the lack of pegmatite maps combined to assert the thesis of heterogeneity and irregularity of mineral distribution in pegmatites.

That some pegmatites are characterized by a well-defined and orderly internal arrangement of petrologic units was, however, early recognized. Hitchcock in his report on the geology of Massachusetts wrote (1833, p. 505), "The most noted locality of green and red tourmalines is in Chesterfield on the land of Mr. Clark. They are contained in an enormous vein of granite in mica slate, which corresponds nearly in direction with the layers of slate. This granite is crossed obliquely by a vein, varying in width from six to eighteen inches, of smoky quartz and silicious (sic) feldspar: or rather the quartz forms the central part of the vein, and the feldspar lies on each side of the quartz: the green red and blue tourmalines, with schorl and sometimes beryl passing through the feldspar and the quartz." It is not entirely clear whether his description deals with primary structures in a pegmatite or with banding in a fracture-controlled unit cutting a larger pegmatitic mass.

A systematic distribution of minerals in the Etta (Black Hills) pegmatite was reported by Blake (1884), and a reproduction of a map by Bailey (Blake, 1884, p. 606) shows a marked concentric structure in the deposit. Shortly thereafter banding was noted in a pegmatite on 110th Street, New York City (Kemp. 1888). Van Hise (1904, p. 724), in writing of the pegmatites of the Black Hills, states, "Still farther away (from the granite) the pegmatitic masses begin to have vein-like characters—that is, there is a rough concentration of the material in different layers parallel to the walls. Still farther away a true banded-vein structure is found."

An interesting description of a zoned pegmatite was presented by Holmquist (1910, p. 804) for a pegmatite of the coast region of Stockholm, Sweden: "Such a pegmatite, traversing Archean limestone, has been worked on the little cliff 'Vånkobben.' It is a dike of about 10 m in

width, composed of pure masses of quartz, microcline and a little graphic feldspar and some pegmatite-granite, which different masses appear in regular zones in the dike. The pegmatite-granite is found as a thin layer or crust on both sides of the wall. After this crust followed inward belts of graphic feldspar of a somewhat irregular thickness. These passed into pure microcline, which form irregular crystals several decimeters in size. The middle of the dike consists of pure quartz, coarse crystalline and a little smoky in colour."

Bastin (1911), Ziegler (1913), and Sterrett (1913) recognized a degree of orderliness in the mineral distribution of the pegmatites they studied, and in the 1920's this phenomenon was reported by Schwartz (1925), Schaller (1925), Schaller and Henderson (1926), and Landes (1928). Kemp (1924, p. 708) stated, "Quartz certainly is the last of all, and in many dikes forms the central part where it seems to be the last residue and to run along the central part like a residual filling." He also noted (p. 709) the existence of banded structures, finer-grained wall zones, and selvages of mica plates normal to the walls. Vogt (1926), p. 229 presented a sketch of a pegmatite from near Arendal, Norway, that shows the quartz core and an arrangement of (mica?) crystals normal to the walls.

The regular distribution of muscovite in pegmatites was summarized by Mohr (1930) in his book, "*Der Nutzglimmer*" (pp. 52-57). Kuznezova (1931) reported that the pegmatites of Western Georgia, U.S.S.R., are zoned between their margins and center. Landes (1932) recognized a coarse-grained core of quartz and microcline in the Baringer Hill, Texas, pegmatite. Hess (1933) noted the existence of large, segregated masses of potash feldspar in many pegmatites. Gevers (1936) emphasized the relation of rare mineral distribution to the contacts of quartz cores in Namaqualand pegmatites.

It was not until 1942, under the stimulation of war-time need for pegmatitic minerals, that the internal structures of pegmatite deposits were systematically studied and the units given map representation. A large number of investigations since that year have indicated that pegmatites with mappable internal units are widespread and that by mapping these units results of both economic and scientific importance can be obtained.¹

Recently there has appeared a short notice on the systematic classification of structural units in pegmatites (Cameron, et al., 1946), and Vlassov (1946) has attempted to classify granite pegmatites on the basis of their internal units.

¹ See for example: Smith and Page (1941); Olson (1942); Pecora (1942); Bannerman (1943); Page, Hanley, and Heinrich (1943); de Almeida, Johnston, et al. (1944); Cameron, Larrabee, et al. (1945); Stoll (1945); Johnston (1945); Griffiths, Heinrich, et al. (1946); Jahns (1946); Shainin (1946A); Cameron and Shainin (1947); and Heinrich (1948).

No matter what detailed version of the origin of pegmatites is ultimately generally accepted, it is certain that two major types of structural units can be recognized in some pegmatites and can be represented accurately on maps and sections. This is not to imply that all pegmatites are marked by a regular internal arrangement of their constituents. However, it has become increasingly recognized in the last seven years that those pegmatites that are of commercial or mineralogical significance generally exhibit this characteristic to a varying degree. The recognition of this systematic distribution of internal elements is of far-reaching significance in the prospecting and mining of pegmatites and in deciphering their genesis.

The two classes of units are (1) primary and (2) secondary. In the following sections definitions of these types are attempted.

Primary Units

Many pegmatite bodies contain well-defined, mappable units of contrasting petrology (i.e., varying mineral content or texture or both) which, in the ideal case, occur as concentric layers around a central unit, or core. Such units, through recent usage, have been termed pegmatite zones (Shainin, 1946B). They are primary in the sense that they represent the original structural elements of the pegmatite body, formed in successive stages of crystallization from the walls inward. The structure and shape of zones reflect the attitude of the pegmatite body as a whole. Not only does their arrangement impart a rough bilateral symmetry in plan but in section as well. Pegmatites containing such units may be termed, *zoned pegmatites*, and those lacking these elements may be referred to as, *unzoned pegmatites*.²

Recent usage has also suggested appropriate names for various zones. The outermost zone along the wall rock contacts is the *border zone*. Because these are commonly thin, they generally cannot be mapped separately, and because they are of relatively little practical importance to the pegmatite miner, they have been ignored. On the other hand some operators have distinguished an economically important *wall zone*, which name can conveniently be retained for the zone adjoining the border zone. The central units are known as *cores*, and a zone immediately surrounding a core is often referred to as a *core-margin zone*. Any zone

² The terms, unzoned pegmatites and zoned pegmatites, are not identical, respectively, with "simple" and "complex" pegmatites as defined by Landes (1933, p. 95). Landes' simple pegmatites, which he defines "as those in which there has been no hydrothermal replacement," may be either unzoned or zoned. His complex pegmatites, defined as those in which "hydrothermal replacement has taken place and rarer minerals have been deposited," probably are all zoned or were zoned originally.

that falls between the core and the wall zone may be termed an *intermediate zone*, of which the core-margin zone is one variety.

The designation of zones in actual practice is not always easy. Lack of pegmatite exposures, changes in relative position of units on different levels owing to the plunge of central and intermediate zones, incomplete development of zones, and disruption and destruction of zones by secondary material are factors that tend to complicate the deciphering of the zonal structure.

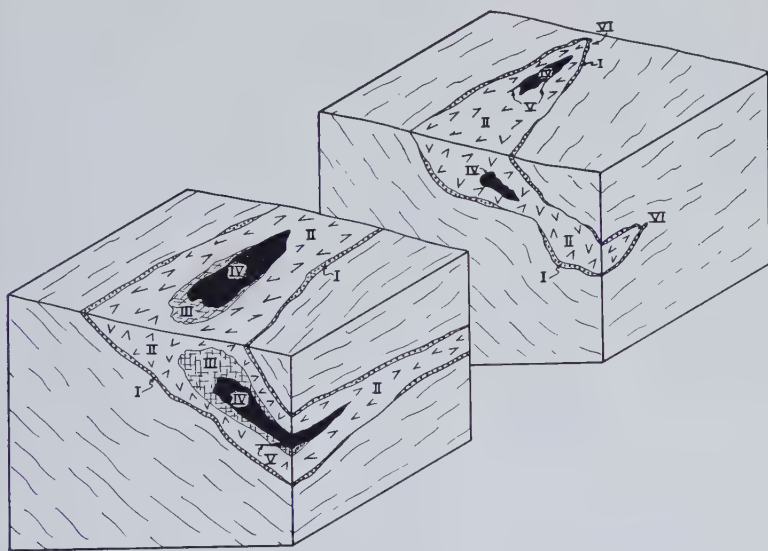


FIG. 7. Idealized expanded block diagram in isometric projection showing pegmatite sill with zonal structure: I—border zone; II—wall zone; III—hood-shaped intermediate zone; IV—core pods; V—transection of intermediate zone by core apophyse; VI—keel of pegmatite, note plunge.

Although, in general, the configuration of zones reflects the over-all shape and attitude of the pegmatite, it should be noted that zones may be imperfectly developed, at least in the horizon under immediate observation. Cores, in particular, may occur as several isolated lenses or pods as well as single central bodies. Some intermediate zones will show a maximum thickness around the end of cores and may pinch out along its sides to form a hood-like unit. Locally within the pegmatite border or wall zones may be discontinuous. These and other features of zones are shown in Fig. 7.

In some flat-lying or moderately-dipping pegmatites the core does not always occur centrally but is displaced toward the hanging wall. Similarly, although a wall zone may be developed along both contacts, one

side may show a difference in mineral content. Thus, a wall zone along the hanging-wall contact may be relatively rich in mica as compared to its footwall counterpart.

A conspicuous feature of zones is that they become increasingly coarse-textured toward the center of the pegmatite. Border zones are usually fine-grained; cores are marked by crystals of gigantic dimensions. There is also a tendency toward simplification in mineralogy toward the core. Outer zones are composed generally of more than two essential constituents; cores are bi- or mono-mineralic.

Obviously the detailed mineral composition of zones varies considerably, depending upon the petrologic type of pegmatite. Wall zones of granitic pegmatites do not contain the same proportions or even the same minerals as wall zones of granodioritic or syenitic pegmatites. Zones in general, however, are composed for the large part of the common rock-forming minerals. Thus zones of granitic pegmatites may contain microcline (or less commonly orthoclase), quartz, muscovite, and biotite as major constituents, and sodic plagioclase, magnetite, garnet, apatite, and schorl as minor constituents. Regardless of the bulk composition, however, zones seem to become more siliceous toward the core. For example, in granitic pegmatites the bulk of plagioclase (primary) may be found in wall zones, the intermediate zones are rich in potash feldspar, and the core is commonly massive quartz. Similarly, in granodioritic pegmatites the outer zones contain a higher proportion of plagioclase, whereas inner zones carry increasing quantities of microcline. There is even evidence that suggests the plagioclase in some of these pegmatites becomes progressively more sodic toward the center of the body. In many zoned syenitic pegmatites wall zones are mafic, rich in an alkali amphibole, whereas cores are felsic in composition.

The number of zones in a pegmatite varies considerably. Within the same district pegmatites may range from unzoned to zoned with three or four units. Pegmatites with more than three zones are not common and those with five are very rare.

The characteristics of primary pegmatite units or zones may be tabulated as follows:

TABLE 3. CHARACTERISTICS OF ZONES

1. Their shape and attitude reflect those of the pegmatite as a whole.
2. Their arrangement gives to the pegmatite a rough bilateral symmetry.
3. The grain size of the constituents increases toward the core.
4. They are composed of the common, rock-forming minerals.
5. They tend to become increasingly simple in mineralogy toward the center.
6. They tend to become more siliceous toward the center.
7. They are few in number; generally there are two or three, uncommonly four, very rarely five.

Secondary Units

Secondary units are those which were formed within crystallized pegmatite by the processes of fissure-filling or replacement, or a combination of the two. They range from thin fracture-filled veins to large irregular masses that represent replacement of a zone or parts of several zones. For convenience there may be distinguished:

1. Veins of fracture-filling.
2. Fracture- and contact-controlled replacement masses.
3. Replacement masses of zones.

Fracture-filled veins are generally too small to be represented on a map although their attitudes may be recorded. Replacement bodies that form outward from fractures will, in their initial stages, be controlled in shape by the fissure. As replacement proceeds the guiding channel tends to be obscured and the tabular bodies grade over into irregular replacement masses. Initial replacement from flat-lying or moderately-dipping fissures favors pegmatite on the hanging-wall side of the crack. In many pegmatites with low-angle dips, the well-defined footwall contact of a massive core with an intermediate or wall zone is a favored

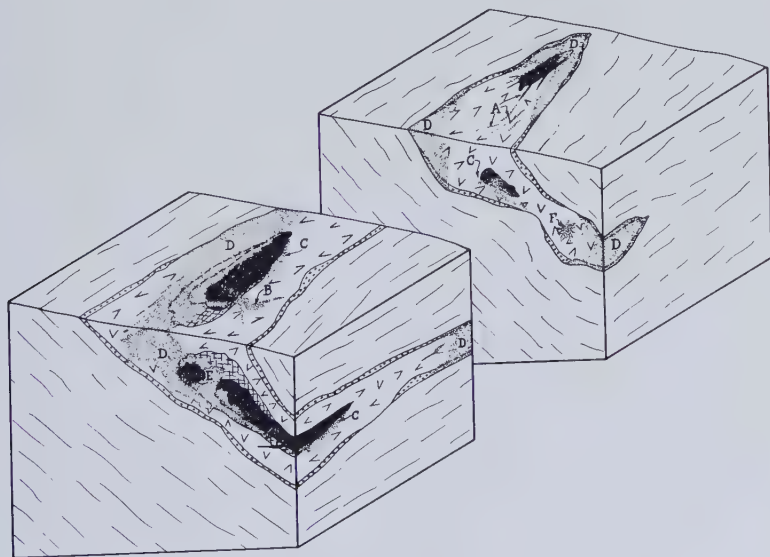


FIG. 8. Idealized expanded block diagram in isometric projection showing pegmatite of Figure 7 with superimposed hydrothermal mineralization: A—filled fractures; B—fracture-controlled replacement vein; C—contact-controlled replacement masses; D—zone replacement masses; E—relict of core and intermediate zone in zone replacement mass; F—radial replacement mass.

channel for localizing replacement. In vertical or more steeply dipping pegmatites both core contacts may show later mineralization. Replacements of a zone or large parts of several zones may themselves show a layered or banded structure, often on a very minute scale. Various types of secondary units are illustrated in Fig. 8.

Secondary units are transgressive and cut across zones and zone contacts. Their shape and attitude do not reflect those of the pegmatite, but are varying and irregular. Their development may destroy the zonal symmetry of the pegmatite. Grain size, texture, and structure are unsystematic and bear no relation to the distance from the contacts. Radial and concentric mineral masses may occur, and corroded and veined relicts of zonal minerals also may be present. Pseudomorphs and vugs of solution are often characteristic.

The rarer mineral species for which some pegmatites are famous are concentrated in the secondary units. In the granitic pegmatites the chief constituents are quartz and sodic plagioclase, commonly sugary albite or cleavelandite. Potash feldspar is rare except as relict material. Lithium micas, alkali tourmalines, and beryl, phosphates, columbium-tantalum minerals, uranium minerals, sulfides, carbonates, zeolites, etc., may be found in these units. Many secondary units may occur in a single pegmatite. For most pegmatites that contain abundant hydrothermal material in secondary units it can be demonstrated that a primary zonal structure is present as well. Unzoned pegmatites containing large or numerous secondary units are not common.

The characteristics of secondary units are enumerated in Table 4:

TABLE 4. CHARACTERISTICS OF SECONDARY UNITS

1. Their shape and attitude are largely independent of those of the pegmatite.
2. They may be cross cutting, and their arrangement tends to destroy zonal symmetry.
3. The grain size is irregular and shows no relation to distance from walls.
4. Corona, rosette and concentric structures, pseudomorphs, relicts, and vugs may be present.
5. Many can be referred to fractures or contacts between zones.
6. The mineralogy is complex, and rare minerals may be abundant. There is no relation of bulk composition to distance from walls.
7. They may be very abundant in any one pegmatite.

INTERIOR PEGMATITES

The interior pegmatites, which tend to be in swarms within the batholith, are tabular crosscutting masses that strike N. 50° W to S. 80° W. and commonly dip 45°–85° NE., and 60°–80° NW. They range in thickness from an inch to six feet but probably average about one or two feet. Some are persistent and can be traced for as much as 700 feet; others are only a few score feet long. They are generally well zoned despite their

small size, and they consist chiefly of microcline, quartz, muscovite, some biotite, traces of black tourmaline, and variable quantities of plagioclase (commonly oligoclase). The microcline is invariably pink to red; the plagioclase is white.

A well-defined swarm occurs just east of the junction of the Mica Lode road and U. S. 50. The dikes, which range in thickness from 1 to $3\frac{1}{2}$ feet, are well differentiated, with six-inch cores of massive quartz and coarse-grained microcline, and outer zones of fine-grained quartz, microcline, and muscovite. One of the group appears to have had a central cleft. Accessory black tourmaline is present.

In the NW. $\frac{1}{4}$, SW. $\frac{1}{4}$, sec. 16 occur several small, well-zoned pegmatites. A section from hanging-wall to footwall shows the following development:

1. Fine-grained light red microcline and quartz; 6 inches.
2. Medium-grained quartz, white oligoclase, and subordinate red microcline; 12 inches.
3. Core of coarse-grained quartz and red microcline; 18 inches.
4. Fine- to medium-grained white oligoclase and quartz; 12 inches.

Just south of this group a pegmatite body, L-shaped in plan, transects a dike of aplite (Fig. 5). The northeastern arm, a sill along the granitic foliation, strikes N. 65° E. and dips 80° NW. However, the northwestern arm, which strikes at nearly right angles to the other and dips 40° NE., appears to have been emplaced along a fracture.

On the north rim of the Gorge, east of the end of the Intake Road, several 6- to 8-inch pegmatites transect reworked xenoliths of hornblende gabbro in Pikes Peak granite. They contain 1- to 2-inch cores of quartz surrounded by zones of fine-grained quartz, red microcline, and white andesine. This is the most calcic plagioclase found in any pegmatite in the area and may be the result of contamination by the mafic host.

A few hundred feet east of this locality on the lip of the Gorge another swarm of small dikes is exposed. One body, which is well exposed on the cliff face, forks with depth and splits into two arms, each of which contains a thin quartz core.

MARGINAL PEGMATITES

All of the larger pegmatites in Pikes Peak granite lie near the contacts with the Idaho Springs formation. These bodies are horizontal to gently dipping, sheet-like or discoidal masses that transect the primary igneous flow structure at nearly right angles. Strikes are very irregular but may be generally to the northwest. Dips, which range from 0° to 25° and average perhaps 10° , are most commonly to the southwest. Changes in thickness and rolls in both contacts are the rule. Included in this group are the largest pegmatites in the district. The Colfelco pegmatite crops out over a length of one mile (northeast-southwest) and is $\frac{1}{4}$ mile wide.

The sheet-like or discoidal shape of the bodies is clearly recognizable. Contacts with the granite are flat-lying or gently dipping, and in many deposits both footwall and hanging wall are exposed. Erosion of rolls in the footwall contacts has resulted in the exposure of the underlying granite in "windows." Some of these pegmatites are roughly circular in outcrop and send off numerous irregular apophyses.

These sheet-like bodies are very poorly differentiated. Zoning, if present, is irregular and ill defined with intergradations rather than clearly defined contacts. The zones tend to be flat-lying, parallel with the contacts. Generally a discontinuous outer zone of varying thickness, consisting of a fine-grained aggregate of quartz, microcline, and muscovite or biotite, lies along both contacts (Fig. 9a).



FIG. 9a. Rounded erosional form, outer zone, Rim pegmatite, in granite.
9b. Unzoned exterior pegmatite sill in schist, MacMullin Ridge.

The bulk of the pegmatite consists of the intermediate zone, a rock composed of two phases. The matrix is a fine- to medium-grained microcline-quartz-muscovite rock in which occur euhedral phenocrysts of graphic granite two inches to two feet across. They resist weathering more than the groundmass and project as knobby, blocky masses (Fig. 10). They consist of a single euhedral microcline crystal as host, with subordinate quartz in an irregular, to subgraphic, to graphic texture. The phenocrysts constitute 10 to 75% of the rock and probably average about 25%. Their cross sections are nearly square and their outlines are as well or better defined than those of the microcline phenocrysts in the Pikes Peak granite. Nearly all of them contain intergrown quartz, but a few of the smaller ones are quartz-free. Like most of the microcline derived from the Pikes Peak magma they are pale pink to deep red in color. Locally red phenocrysts occur in a matrix of white microcline,

quartz, and muscovite, which imparts an unusual mottled appearance to the rock.

Flat-lying pods of massive white quartz or of blocky microcline and quartz form the cores. They are small for the size of pegmatite bodies and are irregularly distributed throughout the central parts. The microcline is free of intergrown quartz and occurs in crystals as much as four or five feet across.

Locally around the margins of the cores occur patches of another distinctive rock type that consists of a subgraphic to graphic intergrowth



FIG. 10. Phenocrysts of graphic granite in Intake Road pegmatite, matrix of fine-grained pegmatite.

of microcline and quartz intermingled in irregular fashion with a quartz-muscovite aggregate. The latter is made up of a dominant quartz host in which are set abundant flakes of muscovite in a semi-regular pattern. The flakes may tend toward parallelism or approach a dendritic or plumose pattern. A few scattered books of muscovite may also occur along the margins of the core. Rarely do they exceed a width of two inches.

With one major exception, fracture-controlled or replacement units are absent or very subordinate. Thin veinlets of black tourmaline and quartz cut across the zones. The School Section pegmatite, the only major exception, contains small replacement units of plagioclase-muscovite pegmatite along the footwall contacts of many of the core pods. Beryl, tourmaline, garnet, triplite, and other rare minerals occur in these units.

A few marginal pegmatites consist chiefly of quartz with subordinate amounts of other minerals. The largest representative of this type occurs southeast of the School Section quarry. This pod-like body which strikes N. 70° E., consists of 80–90% massive white quartz and very minor amounts of microcline, biotite, and muscovite. Across the valley from the Mica Lode quarry is exposed a quartz-rich pod, 30 feet long and 6 feet wide, with narrow stringers and small patches of microcline. At the mouth of Catlin Gulch along the schist-granite contact lies a mass of pegmatite about 250 feet long that strikes N. 56° E. and dips 65° SE. It is highly quartzose and fractured and has been stained by iron oxides. Abundant small masses and veins of fine-grained oligoclase-quartz-muscovite rock occur in it. Along the schist wall is a selvage of 2-inch muscovite books that tend to be normal to the contact. These pegmatites appear to be similar to the “feldspathized quartz veins” in the Archean of the Grand Canyon, Arizona, as described by Campbell (1937, pp. 441–444).

EXTERIOR PEGMATITES

The Idaho Springs schists have been intruded conformably by numerous bodies of pegmatite. The larger masses are close to the Pikes Peak granite contact, but smaller pegmatites occur throughout the entire belt. The pegmatites range in thickness from a few inches to 800 feet and in length from several feet to 3,000 feet. The larger, more irregular masses tend to be concordant but extend numerous apophyses across the foliation. Tabular and pod-like shapes are most common. The strikes range from N. 55–85° E., with generally steep to moderate dips to the northwest. An exception to this general shape and attitude is the Border Feldspar No. 1 pegmatite, which is a very irregular flat-lying lens with offshoots from its crest. Another unusual pegmatite is the Lorain deposit, which is L-shaped in plan.

The intrusion of pegmatite was accompanied by minor wall rock alteration. Mica of the schists has been recrystallized or coarsened, and around some of the smaller sills tourmaline metacrysts have been formed. Some hornblende gabbro has been altered to a muscovite-rich rock by the intrusions.

Many of the larger bodies contain a characteristic zonal structure. Border zones, which are normally narrow and discontinuous, consist either of quartz with small disseminated flakes of muscovite or of coarse books and flakes of muscovite, closely intergrown and normal to the walls. Wall zones may be strongly developed and are composed of a fine-grained, uniform intergrowth of quartz, microcline, and muscovite.

The greater mass of most of the deposits is made up of the intermediate

zones. In mineralogy they resemble their counterparts in the marginal pegmatites, but the two differ markedly in texture. Two phases are present: an irregular to graphic intergrowth of quartz and red microcline, and a quartz-muscovite rock in which the mica flakes tend toward parallelism or dendritic and plumose patterns. These two distinct phases are intergrown in extremely irregular fashion. Although the proportions vary somewhat, neither predominates strongly. Contact relations between the two rock types are confused and contradictory, and their periods of formation probably overlapped. In some exposures the quartz-mica rock appears to penetrate and replace graphic granite. Elsewhere the latter appears to be the younger phase. The rock grades into the fine-grained, uniform pegmatite of the wall zones.

Cores are composed of massive white quartz with variable amounts of quartz-free microcline in crystals as much as six feet in width. Locally, with a decrease in the quartz-mica phase, intermediate-zone pegmatite passes into microcline-rich "embryonic" cores in which graphic granite becomes subordinate to blocky, quartz-free microcline. Cores may consist of several unconnected segments as in the Meyers Quarry pegmatite, or they may be a single unit as in the Mica Lode.

Replacement units, which are superimposed on the zonal structure, are relatively strongly developed in many of the larger pegmatites in schist. Most of the units are clearly related to fractures or to footwall contacts between cores and underlying intermediate zones. Plagioclase, either oligoclase or albite, is the dominant mineral with quartz and muscovite next in importance. Locally cleavelandite is important. Muscovite occurs in two ways: as aggregates of tightly intergrown, $\frac{1}{4}$ - to 2-inch flakes ("ball mica"); and as long, wedge-shaped blades arranged in comb structure. Other minerals are garnet, black tourmaline, apatite, beryl, triplite, lepidolite, red and green tourmaline, and chalcocite.

The structural elements described above, with variable development of the replacement units, are characteristic of the following exterior pegmatites: Meyers Quarry, Mica Lode, Suzana Nos. 1, 3, and 4, Border Feldspar Nos. 1 and 2, Magnusson Crosscut, as well as other smaller bodies.

Most of the atypically zoned Lorain pegmatite is a poorly differentiated aggregate of microcline and quartz, but the thicker part of the northwest limb contains a microcline-rich core. Massive quartz is almost entirely absent. Finely banded pegmatite occurs in the Suzana No. 5 deposit. The bands form swirls, scallops, and irregular curved patterns (Fig. 11). They appear to represent reworked masses of schist.

Many of the smaller pegmatites (one to six feet in thickness) are poorly zoned or completely undifferentiated (Fig. 9b). Some consist of a fine-



FIG. 11. Banded, fine-grained pegmatite, Suzana No. 5 pegmatite.

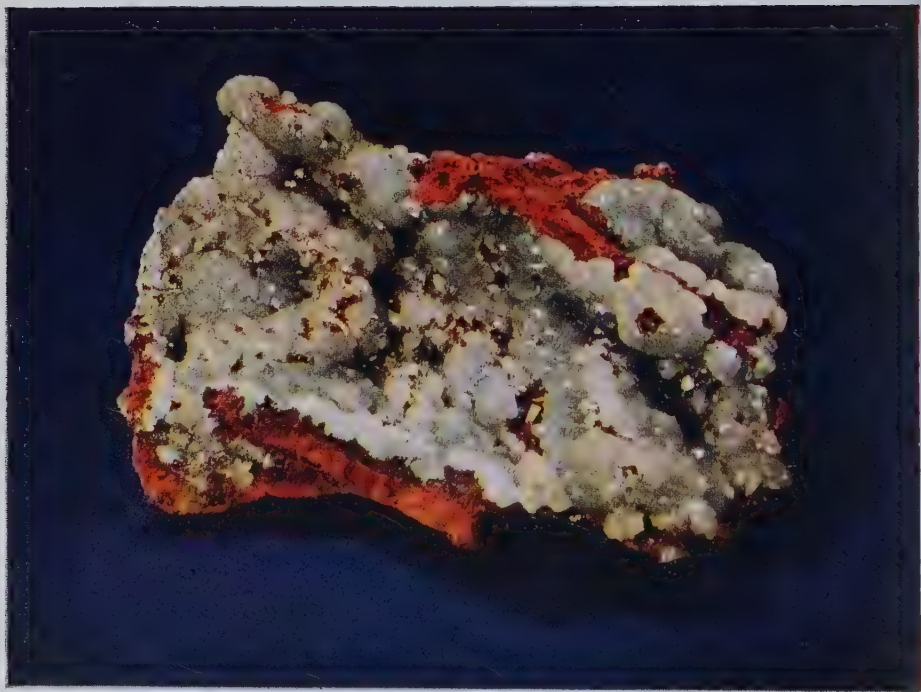
to medium-grained mixture of microcline, quartz, and muscovite. In the main tributary of Catlin Gulch there occurs a type which consists of a fine-grained matrix of quartz and microcline stippled by $\frac{1}{2}$ -inch crystals of muscovite.

Near the contacts with injection gneiss the pegmatites contain increasingly important quantities of magnetite, which occurs as larger euhedral crystals scattered through a fine- to medium-grained aggregate of microcline and quartz.

Near the sedimentary hogbacks south of the Suzana No. 5 body are exposed a number of 1- to 2-foot pegmatites that consist chiefly of quartz and muscovite and minor microcline. The mica flakes are parallel with the contacts and with abundant elongated wisps of schist. These sills may have formed by the accretion of adjoining lit-par-lit injections through gradual replacement of the intervening metamorphic rock.

Some of the smaller of the zoned pegmatites contain only two units: a core of massive white quartz and an outer zone of abundant muscovite books normal to the walls.

(To be concluded in the September-October issue)



*Photographed by George Karger
from the Harvard Collection*

ADAMITE FROM THE OJUELA MINE, MAPIMI, MEXICO. (Two-fifths natural size.)

ADAMITE FROM THE OJUELA MINE,
MAPIMI, MEXICO*

MARY E. MROSE¹

WITH NOTES ON THE OCCURRENCE BY
DAN E. MAYERS² AND FRANCIS A. WISE³

ABSTRACT

Adamite of unusually pure composition is described from a new locality, the Ojuela mine at Mapimi, Durango, Mexico. A chemical analysis is given together with measurements of the morphological axial ratio ($a:b:c=0.9753:1:0.7055$), the unit cell dimensions ($a_0=8.30$, $b_0=8.51$, $c_0=6.04$ Å), the specific gravity (4.435) and optical constants ($X=1.722$, $Y=1.742$, $Z=1.763$, $2V$ (meas.) $88^\circ \pm 2^\circ$). Analysis gave ZnO 56.78, As_2O_5 38.96, SiO_2 0.26, H_2O 3.53; total 99.53. Spectrographic analysis also revealed Cu (~ 0.1), Pb (~ 0.1), Fe (0.01–0.1), Mg (0.01–0.1), Al (~ 0.01), Ca (~ 0.01), Ag (0.001–0.01) and Ga (~ 0.001).

CRYSTALLOGRAPHY

Most of the crystals of Mapimi adamite occur merged together as radiating crusts or as fan-shaped rosettes on a matrix of limonite. A few

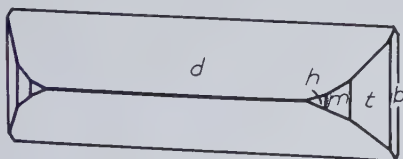


FIG. 1. Adamite crystal from Mapimi showing all the forms observed.

single-crystals, however, were found. The crystals are elongated parallel to [010]. Most crystals were attached at one end of the b -axis; only three doubly terminated crystals could be found. These indicated holohedral symmetry. A test for piezoelectricity by the cathode-ray oscilloscope method gave negative results. The crystals range in size from $\frac{1}{2}$ mm. to 8 mm. along [010].

The crystals are rather simple in habit. The form $d\{101\}$ is dominant and is truncated by $t\{120\}$ and $m\{110\}$. On some crystals the prism $h\{210\}$ occurs, but only as very small faces which gave poor signals during goniometric measurement, and $b\{010\}$ was observed in several instances. The faces in the [010] zone are highly striated parallel to [010], producing a train of signals which made accurate measurements impos-

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sible. A somewhat idealized drawing showing all of the forms observed is given in Fig. 1.

Although a great number of crystals were carefully examined, only nine of the very best of these were selected for measurement (Table 1).

TABLE 1. ADAMITE: MEASURED AND CALCULATED ANGLES

Forms	No. of Xls	No. of Meas.	Size	Quality*	Measured Range		Weighted Mean		Calculated	
					ϕ_2	ρ_2	ϕ_2	ρ_2	ϕ_2	ρ_2
<i>b</i> 010	1	1	VS	C	—	—	—	0°00'	—	0°00'
<i>t</i> 120	9	18	M	B	—	26°52'–27°33'	0°00'	27 08½	0°00'	27 08½
<i>m</i> 110	9	18	S	D	—	45°11'–46 32	0 00	45 44	0 00	45 43
<i>h</i> 210	9	14	VS	E	—	63 42–64 14	0 00	64 01	0 00	64 00½
<i>d</i> 101	9	36	L	B	53°22'–54°59'	—	54 07	90 00	54 07	90 00

* A equals first quality.

The linear elements derived from these measurements ($a:b:c=0.9753:1:0.7056$) are identical with those derived from x-ray Weissenberg measurements on the same crystals ($a:b:c=0.9753:1:0.7055$).

A number of other investigators have reported elements for adamite from other localities and these are listed in Table 2.

TABLE 2. REPORTED MORPHOLOGICAL ELEMENTS OF ADAMITE

Investigator	Locality	Axial Ratio
1. Des Cloizeau (1878)	Chañarcillo, Chile	$a:b:c=0.9733:1:0.7158$
2. Aloisi (1909)	Mte. Valerio, Italy	$a:b:c=0.9736:1:0.7013$
3. Staples (1935)	Gold Hill, Utah	$a:b:c=0.9742:1:0.7095$
4. Mrose (1948)	Mapimi, Mexico	$a:b:c=0.9753:1:0.7055$
5. Rosický (1910)	Thasos, Turkey	$a:b:c=0.9764:1:0.7049$
6. Ungemach (1921)	Laurium, Greece	$a:b:c=0.9770:1:0.7124$
7. Des Cloizeau and Lacroix (1910)	Laurium, Greece	$a:b:c=0.9784:1:0.7117$
8. Kukhareno (1939)	Northwestern Balkhash region (lilac variety)	$a:b:c=0.9787:1:0.7128$
9. Palache (priv. comm.; 1921)	Laurium, Greece	$a:b:c=0.9812:1:0.7108$
10. Laspeyres (1878)	Laurium (emerald-green; cuprian)	$a:b:c=0.9958:1:0.6848$
	Laurium (colorless)	$a:b:c=0.9958:1:0.7176$
11. Murdoch (1936)	Chloride Cliff, Calif. (colorless)	$a:b:c=0.996 :1:0.718$

The wide variation observed appears to be due largely to the poor quality of the measured crystals. In part, however, the differences may be due to variation in chemical composition such as substitution of Cu or Co for Zn or of (PO_4) for (AsO_4) , but no definite correlations could be

established. Laspeyres (1878) has shown that the angles of the adamite crystals from Laurium vary measurably with the presence of Cu in substitution for Zn. The arithmetical average of the linear elements reported, after eliminating those of Laspeyres (1878), Murdoch (1936) and Des Cloizeau (1878), which are relatively far out of line with the others, is $a:b:c=0.9769:1:0.7086$. An angle-table for the forms observed on the Mapimi crystals calculated for these elements is given in Table 3.

TABLE 3. ADAMITE: ANGLE TABLE

Orthorhombic; dipyramidal— $2/m\ 2/m\ 2/m$						
$a:b:c=0.9769:1:0.7086$; $p_0:q_0:r_0=0.7254:0.7086:1$ $q_1:r_1:p_1=0.9769:1.3786:1$; $r_2:p_2:q_2=1.4112:1.0236:1$						
Forms:	ϕ	$\rho=C$	ϕ_1	$\rho_1=A$	ϕ_2	$\rho_2=B$
<i>b</i> 010	0°00'	90°00'	90°00'	90°00'	—	0°00'
<i>t</i> 120	27 06½	90 00	90 00	62 53½	0°00'	27 06½
<i>m</i> 110	45 40½	90 00	90 00	44 19½	0 00	45 40½
<i>h</i> 210	63 58	90 00	90 00	26 02	0 00	63 58
<i>d</i> 101	90 00	35 57½	0 00	54 02½	54 02½	90 00

PHYSICAL PROPERTIES

Cleavage {101} good. The {010} cleavage reported by Staples (1935) on crystals from Gold Hill, Utah, was not observed. Fracture uneven. Brittle. Hardness $3\frac{1}{2}$. $G.=4.435\pm0.005$ (an average of measurements of 8 different crystals on the microbalance); 4.435 (calculated). The specific gravity found for the Mapimi adamite, which is essentially pure $Zn_2(AsO_4)OH$ is comparable to the values 4.475 and 4.484 reported by de Schulten (1903) for the pure artificial compound and by Rosický (1910) for pure adamite from Thasos, Turkey. Lower values, ranging between 4.319 and 4.35, have been found for material containing Cu, Co or Fe'' in substitution for Zn. Luster vitreous. Small crystals on the specimens are nearly colorless, while the larger crystals are greenish-yellow; all are transparent. The Mapimi adamite fluoresces a bright lemon yellow with a pale lemon yellow phosphorescence in short-wave ultraviolet radiation; a much weaker reaction is given in long-wave ultraviolet and in x-rays (pale bluish-green). Specimens from other localities react variously. A blue-green Laurium specimen gave a weak greenish fluorescence in both long- and short-wave ultraviolet; bluish-green, in x-rays. Negative results were obtained with a colorless specimen from Laurium, a blue-green specimen from Gold Hill, Utah, cobaltian material from Cap Garonne, France, and Tsumeb, Africa, and with cuprian material from Tsumeb.

TABLE 4. COMPARISON OF SOME PHYSICAL AND CHEMICAL PROPERTIES OF ADAMITE FROM SEVERAL LOCALITIES

Observer: Mapimi (Mrose, 1948)		Tsumeb, S.W. Africa (Mrose, 1948) (cuprian)	Gold Hill, Utah (Staples, 1935)	Chloride Cliff, Calif. (Murdoch, 1936)	Laurium (Larsen, 1921) (Larsen, 1934)	Northwestern Balkhash Region, (Kukhareenko, 1939)
Color	greenish-yellow	sea-green	rose-red	nearly colorless to pale honey yellow	colorless	lilac
Sign	(+); partly (-)	(-); partly (+)	(+)	(+)	(-)	(-)
Indices:						
α	1.722 $\pm$ 0.003	1.742 $\pm$ 0.006	1.722 $\pm$ 0.003	1.711 $\pm$ 0.003	1.708 $\pm$ 0.003	1.710
β	1.742 $\pm$ 0.003	1.768 $\pm$ 0.006	1.738 $\pm$ 0.003	1.732 $\pm$ 0.003	1.744 $\pm$ 0.003	1.713
γ	1.763 $\pm$ 0.003	1.773 $\pm$ 0.006	1.761 $\pm$ 0.003	1.756 $\pm$ 0.003	1.734 $\pm$ 0.003	1.738
Opt. Orient.	$X = a, Z = b$	$X = a, Z = b$	$X = a, Z = b$	$X = a, Z = b$	$X = a, Z = b$	1.763
Dispersion	$r < v$, strong	$r > v$, strong	$r < v$, strong	$r > v$, strong	$r > v$, strong	1.758
Pleochroism:						
X		nearly colorless	pale rose			pink
Y		bluish green	pale rose			pale rose
Z		yellow green	pale purple			colorless
2V (meas.)	87°-90°	10°-20°	87°-90°	90° $\pm$	87° $\pm$ 05°	86° $\pm$
2V (calc.)	89°37'	23°28'	88°52'	87°17'	86°28'	88° $\pm$
						82°31'
						84°11'

OPTICAL PROPERTIES

The optical properties of the Mapimi adamite are cited in Table 4 in comparison with those reported on material from other localities. The wide variation in the indices of refraction is due to variation in the chemical composition of the crystals, as is clearly shown by comparison of the data on the cuprian and cobaltian material from Tsumeb, but since chemical analyses are lacking in most instances the nature of the relationship is not clear.

X-RAY STUDY

A selected single-crystal of the Mapimi adamite was studied by the Weissenberg x -ray method. The unit cell dimensions, given in Table 5, are in close agreement with those obtained by Strunz (1936) and Kokkoros (1937) on adamite from Thasos and Laurium. The Weissenberg films exhibited orthorhombic centro-symmetry. If the crystal class is

TABLE 5. UNIT CELL DIMENSIONS OF ADAMITE

	1 Adamite (Mapimi) (Mrose, 1948)	2 Adamite (Thasos) (Strunz, 1936)	3 Adamite (Laurium) (Kokkoros, 1937)
a_0	8.30 ± 0.02	8.32	8.31
b_0	8.51 ± 0.02	8.54	8.51
c_0	6.04 ± 0.02	6.08	6.06
$a_0:b_0:c_0$	0.975:1:0.706	0.974:1:0.712	0.977:1:0.712
a_0/c_0	1.38	1.37	1.37
V_0	427	431	429
Space group	$Pnnm$	$Pnnm$	$Pnnm$

taken to be dipyramidal, as is indicated by the form development (summarized by Ungemach (1921)) and by the absence of piezoelectricity, the space group is established by the diffraction effects as $Pnnm$ in conformance with that earlier reported.

The spacing data obtained from a powder photograph of the Mapimi material are listed in Table 6. Photographs of the adamite pattern and of the isostructural species olivenite, $\text{Cu}_2(\text{AsO}_4)(\text{OH})$, and libethenite, $\text{Cu}_2(\text{PO}_4)(\text{OH})$, are given by Richmond (1940). Powder photographs taken of unanalyzed cobaltian and cuprian adamite from Tsumeb were found to differ very slightly in the relative spacing of the lines from that of the Mapimi material and to have very slightly smaller cells.

CHEMISTRY

A chemical analysis of the Mapimi adamite, cited in column 2 of Table 7, proves it to be essentially pure $\text{Zn}_2(\text{AsO}_4)(\text{OH})$. A semi-quantitative

TABLE 6. X-RAY POWDER DATA ON ANALYZED ADAMITE FROM MAPIMI
Copper radiation, nickel filter

No.	<i>I</i>	<i>d</i> (meas.)	No.	<i>I</i>	<i>d</i> (meas.)	No.	<i>I</i>	<i>d</i> (meas.)
1	6	5.944	13	5	2.422	25	3	1.658
2	0.5	5.479	14	4	2.359	26	1	1.622
3	9	4.897	15	1	2.072	27	8	1.608
4	6	4.242	16	1	2.016	28	3	1.581
5	4	3.782	17	3	1.958	29	3	1.570
6	2	3.058	18	2	1.912	30	4	1.511
7	9	2.967	19	2	1.888	31	4	1.483
8	8	2.698	20	1	1.851	32	0.5	1.462
9	4	2.641	21	1	1.811	33	0.5	1.434
10	3	2.582	22	1	1.778	34	0.5	1.408
11	3	2.524	23	1	1.738	35	1	1.395
12	10	2.448	24	2	1.708	36	1	1.377

TABLE 7. CHEMICAL ANALYSES OF ADAMITE

	1	2	3	4	5	6
ZnO	56.77	56.78	31.85	49.11	54.32	54.90
CuO			23.45	1.75		tr.
CoO			0.52	5.16		
FeO					1.48	
MnO						0.11
Rare earths						0.39
As ₂ O ₅	40.09	38.96	39.85	39.24	39.95	39.80
H ₂ O	3.14	3.53	3.68	4.25	4.55	3.45
Rem.		0.26				0.69
Total	100.00	99.53	100.22	99.51	100.30	99.34
G.		4.435		4.352	4.338	4.319

1. Zn₂(AsO₄)(OH). 2. Mapimi, Mexico. McClean analyst, 1947. Rem. is SiO₂. 3. Cap Garonne, France (cuprian adamite). Pisani (1870). 4. Cap Garonne, France (cobaltian adamite). Damour (1868). 5. Chañarcillo, Chile. Friedel (1866). 6. Balkhash region, USSR. Rem. is SiO₂. Kukharenko (1939).

spectrographic analysis, however, shows that other elements are present in significant amounts: Cu~0.1, Pb~0.1, Fe 0.01–0.1, Mg 0.01–0.1, Al~0.01, Ca~0.01, Ag 0.001–0.01, Ga~0.001.

Of the 11 analyses of adamite reported in the literature,¹ only that of the material from Thasos, Turkey, is comparable in purity to the Mapimi mineral. The axial ratio and specific gravity of the crystals from these two localities also are more closely related to each other than to material

¹ Ten analyses in Doelter (1918); with a more recent analysis by Kukharenko (1939).

from other localities. The other reported analyses of adamite all show various amounts of Cu, Co or Fe'' in substitutional solid solution, and small amounts of Mn'' and rare-earths also have been reported. Substitution of P for As has not been reported although this occurs in some related minerals, notably conichalcite, $\text{CaCu}(\text{AsO}_4)(\text{OH})$. The analyses showing the maximum amounts of these substituting elements so far reported are cited in Table 7, and the atomic ratios of the cations corresponding to these analyses are given below. Sufficient data are not at hand to permit a definitive statement of the relation between

cuprian adamite (analysis 3) Cu:Zn=1:1.33

cobaltian adamite (analysis 4) Co:Zn=1:8.8

ferroan adamite (analysis 5) Fe:Zn=1:32.4

the variation in composition and the concomitant variation in optical properties and specific gravity.

ACKNOWLEDGMENTS

The writer wishes to take this opportunity to express her gratitude to Dr. Clifford Frondel and Dr. C. Wroe Wolfe for valuable suggestions and advice during the preparation of this paper; also to Mr. Dan Mayers and Mr. Francis Wise for material on which this study was made, and for providing the chemical analysis.

NOTES ON THE OCCURRENCE

The Ojuela Mine is located at the base of the northeast escarpment of the Sierra de Mapimi, about five miles southeast of the town of Mapimi, Durango. It is one of Mexico's largest mines, and one may spend weeks travelling underground without revisiting the same spot. The mine has been worked to shallow depths for rich secondary silver ores since 1600. In 1893 it was converted into an enormous lead-silver property by the American Metals Company and almost four million tons of ore, assaying on the average 3.7 grams gold, 462 grams silver and 14.9 per cent lead have since been mined. Large scale operations ceased a decade ago in the face of mounting drainage difficulties and depletion of reserves, and work is now carried forward by a mining cooperative.

The Ojuela deposit is a limestone replacement, typical of northern Mexico (Hayward (1931) and Foshag (1934)). Mineralization is controlled by a system of fractures and favors certain dolomitic horizons. Unlike other similar deposits the Ojuela Mine shows signs of igneous activity in its lower workings, small pegmatite veins being present. Enormous caves and pipes, widely separated by barren limestone, contain the principal ore bodies.

In general, the mineralogy of the Ojuela Mine resembles that of Tintic, Utah; most of the species of one locality are represented at the other, although copper minerals occur less abundantly at Ojuela. Primary mineralization emplaced extensive sulfide bodies composed chiefly of arsenopyrite, pyrite, sphalerite and argentiferous galena. Calcite, quartz, fluorite and limonite are the important gangue minerals. A wide variety of minerals resulted from intensive oxidation, among them the arsenates for which the locality is noted. Unfortunately, many of the rarer arsenates, including carminite, scorodite, arseniosiderite, and dussertite have been encountered only sparingly in dump material. A detailed description of these minerals has been given by Foshag (1937).

In June, 1946, the writers had the good fortune to discover a remarkable pocket of adamite in a small manway of the Las Palomas ore body, just above the 11th level. Enroute to a stope containing fine specimens of wulfenite and green mimetite, our lamps fell upon a pocket in the limestone. We saw a miniature grotto, some four feet in diameter and as many deep, its interior carved into fantastic shapes and the entire surface covered with smoothly undulating waves of sparkling yellow crystals; it was a glorious sight, as though we were gazing upon a mineral specimen of unimaginable splendor! Although we could hardly credit our good luck, we set miners to work immediately and obtained some extraordinary specimens. The largest weighed 75 pounds underground, and was almost 3 feet square; it exhibited a continuous crust of green crystals about $\frac{1}{4}$ inch in size resting on a matrix of brown limonite. This specimen, after being trimmed in shape, now rests in the U. S. National Museum. Two other notable specimens are contained in the Harvard collection.

Crystals of adamite from this pocket range up to 5/16" in length. The color varies from water-white through yellow-green (the predominant color) to a pale bluish-yellow. Smaller pockets of adamite subsequently found in the immediate vicinity contain crystals of a paler yellow color. In addition to the adamite found underground a few specimens were collected from surface dumps near the Norte and America No. 1 shafts. These specimens were of poor quality but occasionally exhibited a pinkish color and were sometimes on a matrix of arseniosiderite, while the matrix of the adamite found underground was merely limonite or limestone.

The particular pocket of adamite which we discovered appears to have resulted from a combination of favorable conditions. It seems likely that descending solutions were zinc-rich, as hemimorphite, occurring in large crystals reminiscent of those found at Santa Eulalia, Chihuahua, is abundant in the Palomas ore body between the 11th and 12th levels; although no hemimorphite was found associated with the adamite, smith-

sonite was sparingly present. The adamite pocket occurred in an elbow of the Las Palomas ore chimney and solutions entering this pocket would tend to stagnate, permitting undisturbed alteration and the growth of large crystals.

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MINERALOGY AND THERMAL BEHAVIOR OF PHOSPHATES; I. MAGNESIUM PYROPHOSPHATE

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ABSTRACT

A solid phase inversion of $\text{Mg}_2\text{P}_2\text{O}_7$ apparently takes place near 68°C . The inversion was detected initially by the thermal expansion behavior and later confirmed by differential thermal analyses and high temperature x -ray patterns. A simplified furnace was used for obtaining x -ray patterns at elevated temperatures. The polymorphic change is of considerable theoretical interest since the inversion takes place with a marked change in volume. Incidental data on the thermal decomposition of $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ have been obtained.

I. INTRODUCTION

The compound $\text{Mg}_2\text{P}_2\text{O}_7$ has long been used for the quantitative determination of magnesium in the field of analytical chemistry (1). The magnesium is precipitated in highly alkaline solution as the hydrated crystal, $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$, calcined at 1100°C ., and weighed as $\text{Mg}_2\text{P}_2\text{O}_7$.

Various mechanisms have been proposed for the course of decomposition of the magnesium-ammonium salt (2). Kiehl and Hardt (3) showed that $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ lost 5 mols of water but no ammonia below 50°C . They postulate the simultaneous decomposition of the monohydrate and of the ammonium salt itself above 60°C . They give the temperatures at which the dissociation pressure is 1 atmosphere as 105° for the hexahydrate, and 250° for the monohydrate. They find the dissociation pressure of the hexahydrate to be about 5 atmospheres at 159° ; the monohydrate will decompose rapidly at this temperature if it is in an open vessel. Rapid heating will destroy this sequence of the decomposition due to the formation of primary ammonium salts, which volatilize slowly at these temperatures and also lose some P_2O_5 . Others (4) have suggested the possibility of hydrates with fractional amounts of mols of water, and the true nature of the process is far from clear.

During the course of a thermal expansion run it was noted that the ignited compound $\text{Mg}_2\text{P}_2\text{O}_7$ first showed a contraction slightly above room temperature before the normal positive movement due to reversible thermal expansion took place. This unusual behavior demanded a more complete examination and location of the inversion temperature by means of thermal expansion and thermal analysis curves and by the change in x -ray patterns of the material as it was heated through the transformation point.

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II. PREPARATION OF THE MATERIAL

The material which was used for measurements of expansion, thermal analyses and x-ray analyses was prepared according to the standard procedure for the determination of magnesium as outlined in Hillebrand and Lundell (1). Small batches of material were made for individual measurements in order to maintain a higher degree of purity in each sample. C.P. magnesium nitrate was dissolved in distilled water, diammonium hydrogen phosphate was added in excess, and then ammonium hydroxide until the precipitation was complete. The precipitate was allowed to stand for twelve hours, then it was filtered and washed thoroughly with dilute ammonium hydroxide. Double precipitations were made in some cases to insure maximum purity and ideal composition of the precipitate. After drying at 110°C ., the material was either used as such to investigate the thermal behavior of the hydrate or calcined to form the pyrophosphate.

III. THERMAL DECOMPOSITION OF $\text{MgNH}_4\text{PO}_4 \cdot \text{XH}_2\text{O}$

A. Thermal analyses

The samples used had been precipitated as described above and dried at 110°C . The drying process leads to a product which can now be referred to as $\text{MgNH}_4\text{PO}_4 \cdot \text{XH}_2\text{O}$. The white powder was passed through a 150 mesh screen before placing in the nickel block for thermal analysis. The apparatus used was of the automatically recording type, the rate of heating (400°C . per hour) was controlled by a Leeds and Northrup

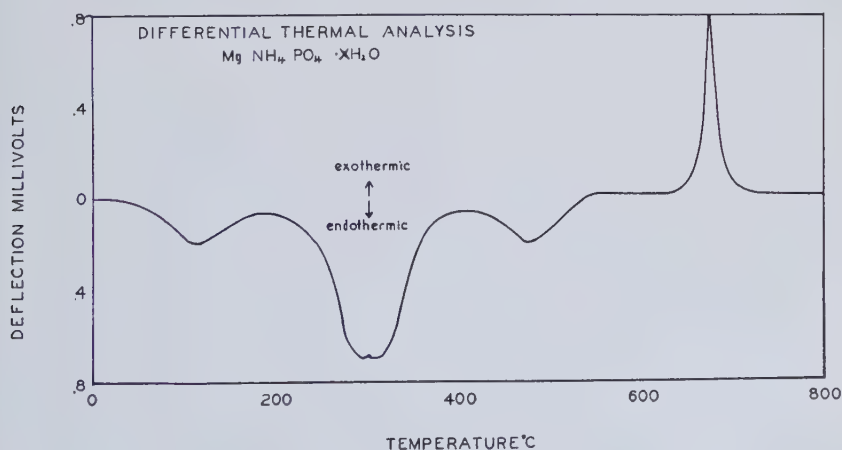


FIG. 1

recording instrument, and the differential peaks were registered by a General Electric photoelectric potentiometer.

During the heating of $\text{MgNH}_4\text{PO}_4 \cdot \text{XH}_2\text{O}$, four principal peaks were observed on the differential thermal curve. The first three peaks were endothermic, the last exothermic. The endothermic peaks occurred in the region $50\text{--}150^\circ\text{C}$., $200\text{--}400^\circ\text{C}$., and $450\text{--}500^\circ\text{C}$., while the exothermic peaks occurred between $650\text{--}700^\circ\text{C}$. The reaction from $200\text{--}400^\circ\text{C}$. was by far the largest of the endothermic peaks, giving a deflection corresponding to 0.7 millivolt at 300°C ., its maximum. The heat effects at $50\text{--}150^\circ\text{C}$. and $450\text{--}500^\circ\text{C}$. were nearly equal in magnitude, having maxima at 110°C . and 475°C ., respectively, with deflection corresponding to 0.2 millivolt at these temperatures. The exothermic peak was very sharp and gave a deflection corresponding to approximately 0.8 millivolt at 675°C . Figure 1 shows an average curve of the differential heat effects.

The endothermic reactions probably signify losses of H_2O and NH_3 while the exothermic peak is apparently due to the heat of formation of the new magnesium pyrophosphate lattice. Samples heated to 600°C . for three hours gave no diffraction pattern indicating that the rearrangement had

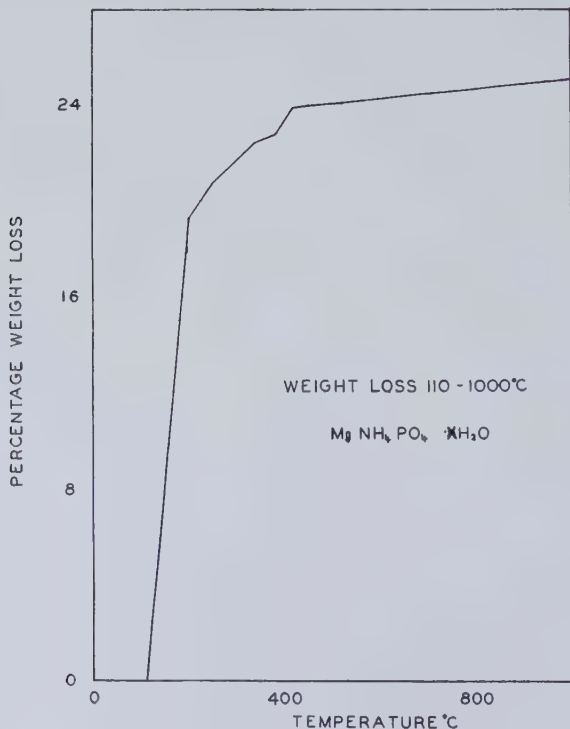


FIG. 2

not yet taken place while other samples heated to 800° C. for eight hours gave the complete pattern of $\text{Mg}_2\text{P}_2\text{O}_7$. These x-ray data then give further evidence for the formation of the pyrophosphate lattice around 675° C.

B. Weight loss measurements

According to the equation, $2\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O} \rightarrow 2\text{NH}_3 \uparrow + 13\text{H}_2\text{O} \uparrow + \text{Mg}_2\text{P}_2\text{O}_7$ a 54.7% weight loss is encountered after complete calcination at 1100° C. Since the thermal decomposition of the hydrate was of only

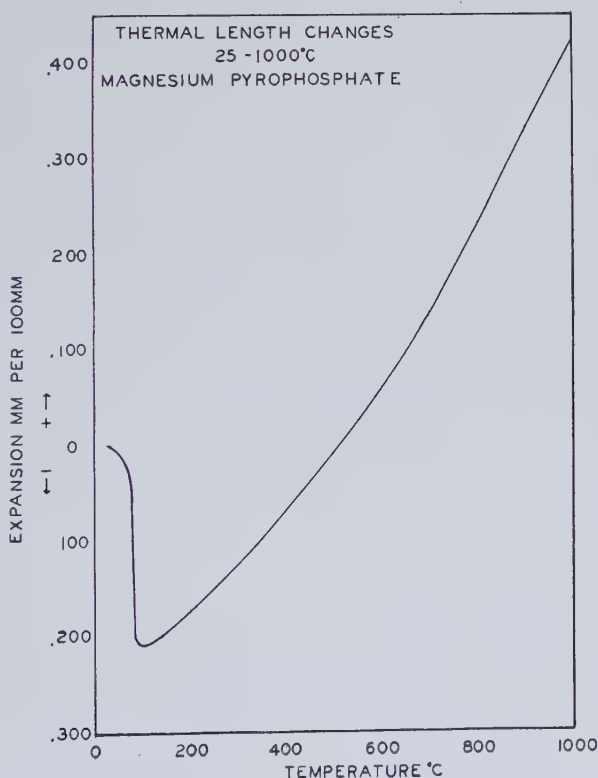


FIG. 3

secondary interest, no attempt was made to determine weight losses which occurred below 110° C. Other weight loss measurements were made between 110° C. and 1000° C.

Figure 2 illustrates average percentage weight losses, over the interval 110°–1000° C. Calculations at each temperature are based on the original weight of the sample dried at 110° C. The greatest loss in weight occurred below 200° C. In the interval 110°–200° C., it was approxi-

mately 19%. Above 200° C. losses in weight were relatively small and amounted to less than 6% in the range 200–1000° C.

IV. SOLID PHASE INVERSION OF $\text{Mg}_2\text{P}_2\text{O}_7$

A. Results of thermal expansion measurements

The samples used for expansion runs were made from magnesium pyrophosphate which had been calcined one hour at 1150° C., passed through a 120 mesh screen, temporarily bonded with a water solution of Carbowax and Methocel, pressed into bar samples approximately $4'' \times \frac{1}{2}'' \times \frac{1}{2}''$ in size, and finally fired for $\frac{1}{2}$ hour at 1320° C. An expansion apparatus utilizing a fused silica sample holder, fused silica transmission rod and dial gauge reading to one ten-thousandth of an inch was used to measure length changes.

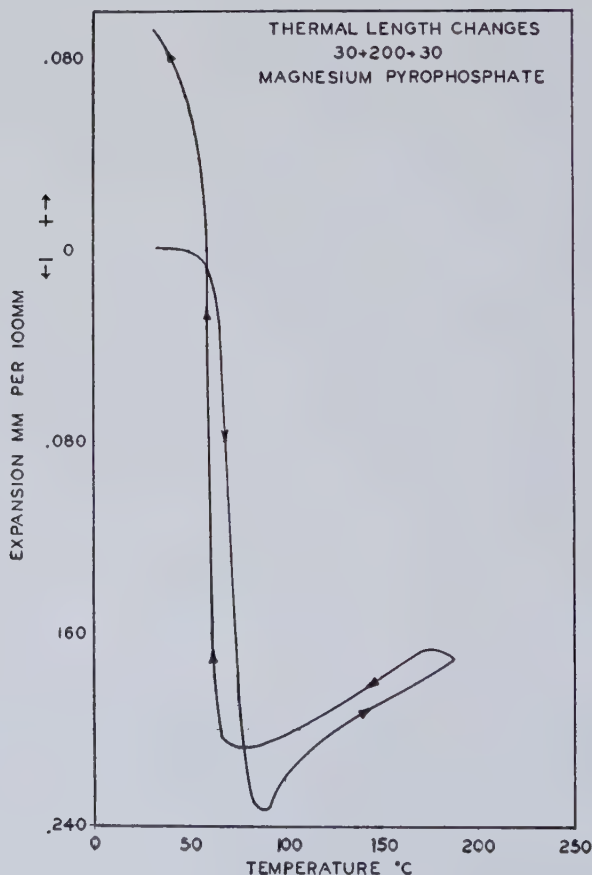


FIG. 4

It was found that in a regular expansion run, while heating at a rate of 100° C. per hour from room temperature to 1000° C., the sample contracted very rapidly from about 50°–90° C. and reached a minimum at about 95° C. At this point, expansion began, and proceeded at a regular rate until the run was completed, at 1000° C. This period of contraction coincides nicely with the temperature region near 68° C., where the inversion from low to high magnesium pyrophosphate takes place, as determined by thermal analyses and x-ray patterns.

Figure 3 shows the expansion in mm./100 mm. plotted against temperature, and illustrates the contraction of the sample during the period 50°–90° C., with normal expansion from 95°–1000° C. The coefficient of expansion of the high temperature form of $\text{Mg}_2\text{P}_2\text{O}_7$ over the range 100° C. to 1000° C. was approximately 70×10^{-7} cm./cm.° C.

Thermal length changes were also measured on a sample which was heated from room temperature to 200° C. and then cooled from 200° C. to room temperature. The rate of heating in these runs was considerably slower (about 60° C. per hour), and magnified curves of thermal expansion and contraction near the inversion temperature were obtained. These curves, as expected, showed rapid contraction between 50–90° C. on heating and a gradual expansion from 95–200° C. On cooling from 200° C., there was a gradual contraction of the sample until 70° C. was reached. Here a very rapid expansion took place, tapering off gradually below 50° C. and leaving the sample with a permanent length change of approximately 0.1%. The solid phase inversion, it would appear, is reversible. The following curve (Fig. 4) shows graphically the results of the expansion-contraction run between room temperature and 200° C.

The fact that the inversion took place within a narrower temperature range on cooling may be due simply to the existence of a more uniform temperature distribution in the sample on cooling as compared with the distribution during the heating cycle. After repeated heating through the inversion range, bar samples showed many cracks over their entire length, giving further physical evidence of the disruptive force of the reversible volume change.

B. Results of thermal analyses

The samples used were magnesium pyrophosphate powder which had been calcined one hour at 1150° C., cooled, then passed through a 150 mesh sieve. The apparatus used was the same as that described in Part A of Section II above.

There was but one peak on the differential heating curve. This peak was endothermic, with a deflection corresponding to about 0.4 millivolt and had its maximum at 68–70° C. The endothermic peak, Fig. 5, represents a change in lattice structure from the low temperature monoclinic

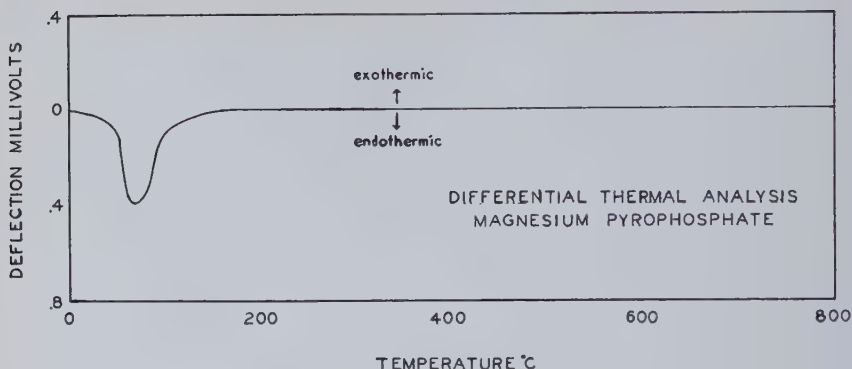


FIG. 5

form usually reported (5) to some high temperature form, the crystal structure of which has not yet been determined.

Further evidence of the reversibility of the inversion was obtained by recording the heat effects shown by the above samples on both heating and cooling. Figure 6 is a plot of these effects.

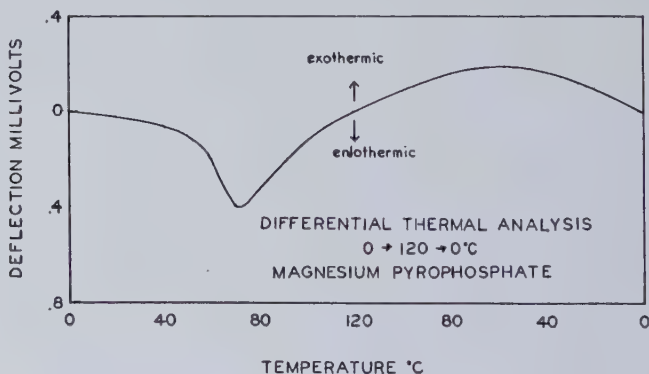


FIG. 6

The height of the endothermic peak was greater than that of the exothermic peak, but the areas under both curves were approximately equal.

C. Results of x-ray analyses

The x-ray data were obtained by the use of two different machines, the first being a General Electric, Model XRD, Type 1, giving iron radiation ($\lambda=1.93 \text{ \AA}$) passed through a beryllium window. Conventional Debye-Scherrer powder photographs were made with a camera of 7.16 cm. radius. The tube was operated at 5 milliamperes and exposures were made for six hours.

Additional x-ray patterns were obtained with a Norelco Geiger-counter x-ray Spectrometer, Type 42322, with $\text{CuK}\alpha$ radiation ($\lambda = 1.537 \text{ \AA}$), filtered through nickel. The peaks from this machine are automatically recorded on a Brown "Electronik" strip chart potentiometer. Ninety minutes were required to traverse the 90° goniometer arc and the tube was operated at 5.7 milliamperes.



FIG. 7

The heating furnace which was used on this latter machine was of simple design and was constructed in the instrument shop of the School. It was similar in many of its essential features to the recently published (6) Bureau of Standards design as can be seen in Figs. 7 and 8.

To test the furnace element one may calibrate it by using known inver-

sions as fixed points, or as shown by Jay (7), by actually measuring the expansion of a known substance.

The expansion of quartz was determined from room temperature to 700° C., and good agreement was obtained with the known values. The



FIG. 8

break in the curve at the inversion temperature (Jay, 7 cit.) was found to be at 575° C. $\pm$ 5. The constancy of temperature over the sample face, as also over the period of time concerned, was $\pm$ 5° C. Standardization in the range of the sample studied was obtained with ammonium nitrate as used by Buerger (8), et al. The inversion temperature obtained was 83° C. which is in excellent agreement with previous values. To deter-

mine the temperature of inversion, x-ray patterns were taken at temperatures above and below the expected temperature, in a regular series. This gave the limits within which the inversion had taken place. To avoid the more tedious process of taking a large number of complete patterns at each of a series of closely spaced temperatures, the following modification was devised: The Geiger-counter arm was fixed at an angle where the phase to be formed has an intense peak corresponding to a reflection. The temperature of the furnace was then raised or lowered very slowly across the range. Since there was not control to insure a uniform rate of rise of temperature, temperatures were read and recorded frequently. The shape of the curve obtained as a record of the intensity of the reflection gives an excellent indication of the temperature of inversion. The two changes in slope and two level areas will be seen to stand out well in Fig. 9, the actual record for NH_4NO_3 .

TABLE 1. SPACINGS FOR LOW AND HIGH TEMPERATURE FORMS OF $\text{Mg}_2\text{P}_2\text{O}_7$

ASTM card catalogue low temperature form		Norelco machine				General Electric machine low temperature form	
		low temperature form, 25° C.		high temperature form, 250° C.			
<i>“d”</i>	<i>Rel. Int.</i>	<i>“d”</i>	<i>Rel. Int.</i>	<i>“d”</i>	<i>Rel. Int.</i>	<i>“d”</i>	<i>Rel. Int.</i>
4.14	.10	4.14	.19	4.175	.2		
3.85	.04	3.85	.07				
3.45	.05	3.465	.09				
		3.15	.09				
3.00	1.0	3.00	1.0	3.01	1.0	2.995	1.0
		2.97	.45	2.92	.35	2.965	.50
2.51	.08	2.52	.14	2.535	.09		
2.40	.05	2.41	.10				
		1.87	.04	1.865	.08		
1.85	.10	1.846	.05	1.828	.08		
2.30	.01						
2.15	.02						
2.09	.15	2.09	.11	2.10	.11		
		1.714	.02	1.72	.04		
1.69	.08	1.70	.06	1.68	.03		
1.57	.10	1.57	.08	1.585	.16	1.57	.30
1.465	.07	1.469	.02	1.48	.05		
1.38	.06	1.385	.02	1.385	.06	1.38	.16
1.322	.07					1.32	.1
1.250	.05						
						1.138	.2
						1.05	.3
						1.011	.1
						1.00	.8

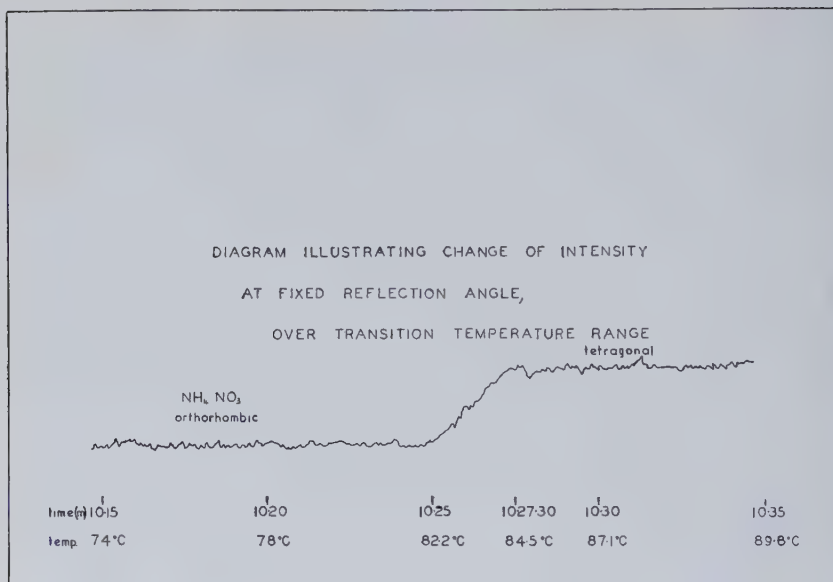


FIG. 9

Table 1 gives the three sets of “*d*” values for the monoclinic low form of $\text{Mg}_2\text{P}_2\text{O}_7$ obtained, (a) with the sample in the furnace on the Norelco machine, (b) by the General Electric machine, (c) from the ASTM manual, each at 25° C. Comparison of the experimental values with the ASTM data shows some definite omissions in the latter, due probably to lack of resolution of some doublets. Complete recordings over the arc 2–90° were run at temperatures as low as 7° C. and also at 20, 40, 60, 80, 100, 250, 450 and 850° C.

A distinct change was noticed between the temperature of 60° C. and 80° C. The main differences may be noticed in the disappearance of the peak at 30° 27' and the appearance of a new peak at 30° 55'. These same peaks were examined at temperatures of 62, 64, 66, 67, 68, 70 and 73° and the appearance of the change is illustrated by Fig. 10, and the temperature of the inversion could therefore be put at $68 \pm 2^\circ$ C. The simultaneous existence of the two peaks, in the light of the immediate low to high reversible type of change admits of two explanations. Firstly, that some of the material at all these temperatures is at a different temperature from the rest, due to air currents and surface cooling; secondly, that the change actually takes place over a very small range of temperature. The former is the more probable explanation although the latter would be very interesting if true.

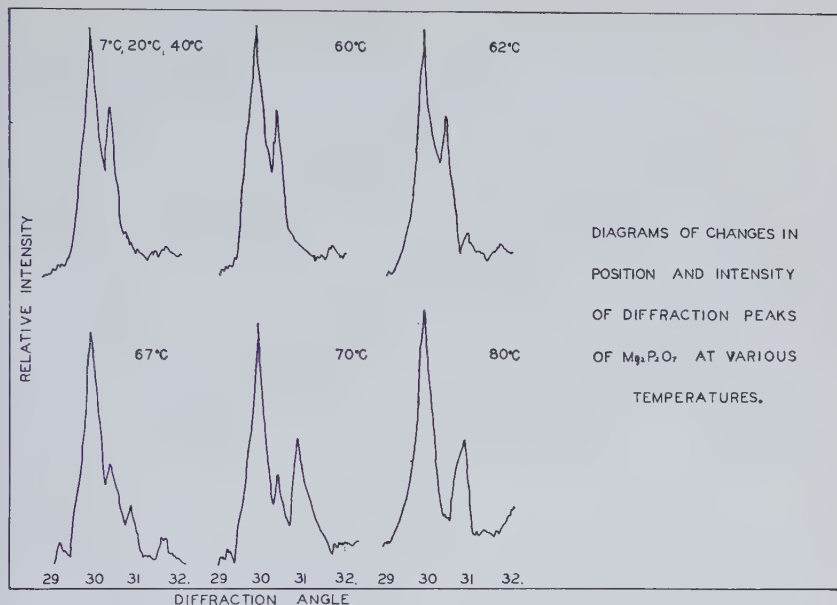


FIG. 10

This rapid and simple method of determining inversions should find wide application. The extension of the upper temperature limit to 1500° C. with an alumina holder and a platinum-wound furnace is a simple step, and the study of high temperature forms, especially in the rapidly reversible type systems, should be much simplified. The chief difficulty lies in the falling of the sample from the holder due to decomposition, or due to inversions accompanied by large volume changes. The remedy for this would appear to be the construction of the Geiger-counter type of instrument with the entire Geiger tube arm and the arc lying in a vertical instead of a horizontal plane, which would then allow the sample to be placed and subsequently heated on a horizontal surface.

Another possible method for high temperature work which would not involve radical changes in the design of the present x-ray machine would be the use of a thin sheet of beryllium metal foil for holding the sample in a "pocket" between the foil and the glass slide.

V. SIGNIFICANCE WITH REGARD TO ISOMORPHISM AND POLYMORPHISM

The investigation of the thermal behavior of $\text{Mn}_2\text{P}_2\text{O}_7$ is planned since this compound has been reported to be isomorphous with $\text{Mg}_2\text{P}_2\text{O}_7$ (9). A study of the behavior of other divalent pyrophosphates of zinc, nickel, cobalt and iron might reveal similar inversions due to the close proximity

of the ionic radii of each of these elements to that of magnesium. Similar inversions for zinc pyrophosphate and copper pyrophosphate have already been detected here, although the ionic radius of copper is considerably larger than that of magnesium.

The fact that these inversion temperatures can be determined quite accurately, that the pyrophosphate group is common to all the compounds, and that the ionic or atomic radii of these divalent cations are well known from independent data may serve to shed some light on the nature of polymorphic changes.

When the structures of the high temperature forms of the pyrophosphates are determined, it may be possible to state whether the change is due to rotating molecular groups, such as has been suggested for ammonium nitrate (10), or whether increased thermal motion causes a partial dislocation of certain atoms similar to the well-known cristobalite transition.

VI. SUMMARY AND CONCLUSIONS

A thermal study of the decomposition of $\text{MgNH}_4\text{PO}_4 \cdot \text{XH}_2\text{O}$ reveals no new data relative to the mechanism and temperature of loss of H_2O and NH_3 .

By means of thermal analyses, thermal expansions, and x -ray data, a solid phase inversion of $\text{Mg}_2\text{P}_2\text{O}_7$ has been placed at approximately 68°C . X -ray data indicate an accuracy of $\pm 2^\circ\text{C}$. for the rapid low to high inversion.

A large volume contraction and an endothermic heat effect attend the change from the low temperature monoclinic form to a high temperature form whose crystal system has not yet been determined.

The density and refractive indices previously reported (11) for $\text{Mg}_2\text{P}_2\text{O}_7$ are apparently for the low temperature monoclinic form, while the melting point data (11) are for the high temperature form.

ACKNOWLEDGMENT

Appreciation is expressed to Dr. T. F. Bates of the Department of Earth Sciences for suggestions concerning the construction of the high temperature x -ray furnace and to Dr. E. C. Henry and Dr. H. M. Davis of the Department of Mineral Technology for encouragement during the course of the work.

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TINCALCONITE CRYSTALS FROM SEARLES LAKE, SAN BERNARDINO COUNTY, CALIFORNIA

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ABSTRACT

The discovery of small, well-formed crystals of tincalconite in drill cores from Searles Lake, San Bernardino County, California, is announced. The associated minerals are described and the results of goniometric and x-ray examination of tincalconite reported.

INTRODUCTION

The name tincalconite first appeared in print in 1878.¹ The statement in which it was contained reads as follows:—

“Tincalconite (Shepard). Borax pulverulent et efflorescent, de Californie, 32p. cent d’eau.”

This appeared in a section of the *Bulletin* headed “Extraits de diverses publications.” Literature references are given for most of the extracts, all of which are concerned with new mineral names, but no reference is given with the extract on tincalconite which is quoted in full above. No trace has been found of any original publication by Shepard² on this material and it seems that such may never have been accomplished.

Mineralogists generally³ considered the name tincalconite to apply to a variety of borax rather than to a distinct mineral. After half a century it was pointed out by Schaller⁴ that “it is obvious” that Shepard’s tincalconite “was the 5-hydrate of the borax series, $\text{Na}_2\text{O} \cdot 2\text{B}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ identical in composition with ‘octahedral borax’.” According to Schaller it is “not very abundant but rather widespread, coating both borax and kernite.” It forms from borax, the 10 hydrate of the series, by partial dehydration or from kernite, the 4 hydrate, by hydration. So far it has been found only in powdery form.

Natural borax crystals such as are found embedded in the muds of Borax Lake in Lake County and Searles Lake in San Bernardino County, California, and numerous other localities are probably partly or wholly altered to the 5-hydrate and so should be considered pseudomorphs of tincalconite after borax. It is very easy to observe the process of partial

¹ *Bull. Soc. Min. France* **I**, 144 (1878).

² No initials are given with this name but it may be presumed that this was Charles Upham Shepard (1804–1886), an American mineralogist who was a most active and not always careful namer of minerals.

³ Dana, E. S., *System of Mineralogy*, 6th ed. 887 (1892); Eakle, A. S. *Minerals of California*, *Bull.* **67**, Calif. State Mining Bureau, 170 (1914).

⁴ Schaller, W. T., *Borate minerals from the Kramer District, Mohave Desert, California: U. S. Geol. Surv. Prof. Paper* **158-I**, 163 (1930).

dehydration in the laboratory. Perfectly clear, well formed crystals of the 10 hydrate are easily grown in the laboratory by evaporation of an aqueous solution at room temperature. If such crystals are left unprotected in a dry atmosphere they become dull, white and opaque within a few days or weeks. In some cases one can observe the whitened part extending from the surface into the clear borax crystal as a small bulbous or frond-like growth. This sort of change can be prevented or arrested by sealing the crystals in a small tube or coating them with shellac. It has been found possible to maintain crystals partly whitened and partly clear in a sealed tube for ten years or more.

DISCOVERY

Last summer one of us (D. L. S.) collected specimens from cores obtained by deep drilling at Searles Lake. One sample of a nine inch core was observed to contain numerous clear crystals suspected of being tincalconite. It was brought into the laboratory for further study and most of this report is based upon work on this sample.

GEOLOGICAL SETTING

Searles Lake is a salt body occupying a large basin in the northern part of San Bernardino County. There is water at the lake surface only in unusually wet seasons. A firm crust of salts covers an oval area of 11 or 12 square miles.⁵ At the margins the salts are mixed with or covered by alluvial material. The total area of the playa zone, composed of salts and mixed alluvial material, is about 60 square miles. The salt body is exceedingly porous, consisting in large part of reticulated crystals with open spaces constituting more than 25%. These open spaces are occupied by a heavy brine which fills the numerous drill holes and is the chief raw material of the extensive chemical industries operated at the edge of the "lake."

According to Gale⁶ the depth of the salt deposit is 60 to more than 100 feet, probably averaging 70 to 75 feet. The deepest of many wells whose records were reported by Gale penetrated salts to a depth of 85 feet. In recent years numerous borings carried to a greater depth have shown that there exists a deeper salt body under the muds that make up the bottom of the exposed salt body. These muds are 10 to 20 feet thick. Below them lies a "second" salt body sometimes referred to as the "lower structure." This lower salt body is some 40 or 50 feet thick.

⁵ Gale, Hoyt S., Salines in the Owens, Searles, and Panamint basins, southeastern California: *U. S. Geol. Surv., Bull.* **580**, 251-323 (1915)

⁶ *Op cit.*, p. 273.

The core sample in which the tincalconite crystals were found was taken from deep well X-1, situated in sec. 23, R43E, T25S, slightly north of the center of the exposed salt body. This well penetrated to the bottom of the lower salt body at a depth of about 118 feet. Below this it penetrated dark muds with traces of various salts to about 121 feet, the limit of the boring. The core sample studied is 6 to 7 centimeters thick in its thickest portion and came from a depth of about 116 feet, that is from within a few feet of the bottom of the lower salt body.

TINCALCONITE CRYSTALS

The clear, colorless tincalconite crystals are scattered as isolated individuals or clusters of two or three through a thin layer, not over 2 centimeters thick, in the middle part of our core sample. The matrix is fine grained white salts not certainly identifiable with the unaided eye. In a few patches the ratio of volume of tincalconite crystals to matrix may be 1:2 or 1:3, but for the most part the ratio is 1:10 or less. The scattered crystals are easily separated from the matrix, often falling out when the core is broken. Not infrequently there is an open space partly surrounding the larger tincalconite crystals.

The great majority of the crystals lie within a rather narrow size range between one and three millimeters in maximum dimension. By weighing two counted lots of crystals it was found that the average weight of a crystal is just under three milligrams. Taking into account the shape and the density (reported below) it may be calculated that the average crystal is just under 2 millimeters from tip to tip. The largest crystals we have removed from the matrix are about 4 millimeters from tip to tip. Recently Mr. H. Stanton Hill⁷ of Pasadena has found a tincalconite crystal that "would probably measure close to 1 cm. were it to be completely removed from its matrix."

The water content of the tincalconite crystals was estimated from the water loss of two small lots of clear crystals, heated to constant weight, care being taken not to drive off any other material by overheating. By a similar procedure it was shown that the common fully whitened borax corresponds closely in composition to tincalconite. For this purpose a large well formed borax crystal embedded in Searles Lake mud, which had been in the dry laboratory air 5 years, was used. The results of these tests, tabulated below, leave no doubt that both materials are essentially the pentahydrate.

	Weight per cent of H ₂ O
Ideal Na ₂ B ₄ O ₇ · 5H ₂ O	30.9
Tincalconite crystals	30.0
Whitened borax	29.8

⁷ Letter to A. Pabst, dated January 7, 1948.

The specific gravity of small clear tincalconite crystals was determined by suspension in bromoform diluted with methyl alcohol and found to be 1.88. This agrees with the value 1.880 reported for "artificial crystals" by Schaller.⁸ The optical properties were found to agree within the limits of error with those reported by Schaller for "artificial tincalconite."

The tincalconite crystals are very constant in habit. They are rhombohedral, invariably showing the forms $c\{0001\}$ and $r\{10\bar{1}1\}$ combined in such a manner as to closely resemble an octahedron. Other forms are lacking or appear only as minute truncations or strips. This habit is similar to that of artificial crystals which were long known as "octahedral borax." Figure 1 shows how close is this similarity. All the inter-

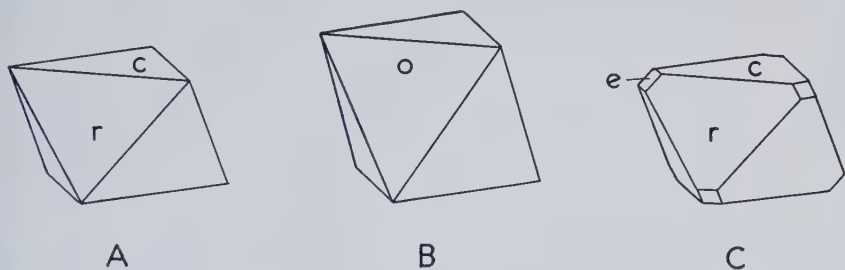


FIG. 1. A. Typical tincalconite crystals from Searles Lake.

$r\{10\bar{1}1\}$, $c\{0001\}$.

B. Ideal octahedron.

$o\{111\}$.

C. $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ grown in laboratory.

$r\{10\bar{1}1\}$, $c\{0001\}$, $e\{01\bar{1}2\}$.

facial angles at the twelve like edges of an ideal octahedron are $70^\circ 32'$. In tincalconite the angle $r \wedge r$ is about $5\frac{1}{2}$ degrees more and the angle $c \wedge r$ is about 5 degrees less. This difference is scarcely discernible on crystals a few millimeters in dimension. On the other hand, all of the faces of an ideal octahedron are equilateral triangles whereas only the c faces of tincalconite are equilateral and the angle between edges of the r faces (this is also the interaxial angle of the rhombohedral axes) is about 12 degrees higher than the corresponding 60 degree angle on an octahedron. This can be noticed by careful inspection on even the smallest crystals.

Crystals of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ were grown in the laboratory from a water solution near 100°C . The small crystals first formed show the same habit as natural crystals but crystals grown to a larger size, 5 to 10 millimeters in maximum dimension, invariably show in addition small faces of the form $e\{10\bar{1}2\}$. With this form present the crystals also appear isometric,

⁸ *Op. cit.*, p. 163.

the new form cutting all of the pseudo-octahedral corners and simulating a cube as shown in Fig. 1C.

Crystals of the pentahydrate were first measured by Arzruni.⁹ He reported the same forms just mentioned and his measurements have been included in Groth's compilation "Chemische Krystallographie."¹⁰ Unfortunately the column headings in Groth's table for calculated and measured angles are erroneous, except that the starred angle is given under the appropriate heading. All references to Arzruni's work herein are to the original publication only.

Four small tincalconite crystals and one laboratory grown crystal were measured on both two-circle and one-circle goniometers. The results showed that even selected natural crystals show such imperfections that precise angle measurements are not possible and that artificial crystals are no better. Arzruni reported variation of angle measurements over two or three degrees but gave values to minutes nevertheless. Table 1 shows a comparison of our results with those of Arzruni. Though no close agreement has been obtained a correction of Arzruni's results is not clearly indicated.

TABLE 1. INTERFACIAL ANGLES OF TINCALCONITE, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

	Average	Range	Number of observations	Arzruni	
				Measured	Calculated
(10 $\bar{1}$ 1 $\wedge$ 1 $\bar{1}$ 01)	103°53'	103°18'–104°31'	12	103°38'	—
(0001 $\wedge$ 10 $\bar{1}$ 1)	65°37'*	64°52'–66°50'	14	62°53'	65 11

* The angle ρ from two-circle measurements. The calculated angle (10 $\bar{1}$ 1 $\wedge$ 1 $\bar{1}$ 01) corresponding to this would be 104°8', whereas the angle ρ calculated from the one-circle measurement of (10 $\bar{1}$ 1 $\wedge$ 1 $\bar{1}$ 01) would be 65°24'.

To check on the axial elements it is fortunately possible to draw upon other data. The cell dimensions of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ have been reported by Minder.¹¹ Table 2 gives axial elements derived from various sources. The agreement between our best values and Minder's best values happens to be fairly close. This should not be taken to indicate a precise determination.

⁹ Arzruni, A., Ueber den sogenannten "octaëdrischen" Borax: *Pogg. Ann. der Physik*, **158**, (234), 250–252 (1876).

¹⁰ Volume 2, page 732, Leipzig (1908).

¹¹ Minder, W., Über den Bau einiger Hydrate von Natriumdiborat: *Zeits. Krist.*, **92**, 301–309 (1935). Minder entitles the section of his paper dealing with the pentahydrate "Tincalconite, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ " suggesting that he may have used natural crystals. Through the courtesy of Dr. Werner Nowacki of Bern, Switzerland, where Minder did his work, it has been possible to ascertain that artificial material was used.

TABLE 2. AXIAL ELEMENTS OF TINCALCONITE, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

	c_h/a_h	a_h	c_h	α	a_r
Arzruni 1876	1.87			$71^\circ 59'$	
<i>op. cit.</i> , p. 251					
Minder 1935	1.850	$11.3 \pm 0.1 \text{ \AA}$	$20.9 \pm 0.1 \text{ \AA}$	$72^\circ 36'*$	9.54^*
<i>op. cit.</i> , p. 305					
Minder 1935	1.887†	$11.20^\dagger$	$21.13^\dagger$	$71^\circ 42' \pm 5'$	$9.56 \pm 0.04 \text{ \AA}$
<i>op. cit.</i> , p. 304					
This paper	1.892			$71^\circ 35'$	
1-circle measure- ment					
This paper	1.911			$71^\circ 8'$	
2-circle measure- ment					

* Rhombohedral constants calculated from Minder's hexagonal cell.

† Hexagonal constants calculated from Minder's rhombohedral cell. Minder gives dimensions as $\text{\AA}$. These should doubtless be interpreted as kX.

As indicated in Table 2 Minder reports separate cell constants for a rhombohedral and for a hexagonal cell, apparently derived from different rotation patterns. It is evident that his constants for the rhombohedral cell are more accurate. If a cell of these dimensions contains $3(\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O})$ the density should be 1.893, only slightly greater than the value 1.88 directly determined on both natural and artificial crystals.

Table 3 shows a record of a powder pattern of tincalconite crystals from Searles Lake made with copper radiation. It may be seen that all observed lines due to spacings over 1.75kX are accounted for and that the agreement of the spacings is such as to give at least a rough check on Minder's cell dimensions. The table also shows the intensities recorded by Minder for certain of these lines in a rotation pattern on the rhombohedron edge. All of the lines he recorded in this range of spacings are found on the powder pattern. There is substantial agreement also with the powder pattern of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ given on card 2233 of the original ASTM card file of powder patterns. The line 3.93 on our pattern is not recorded on the ASTM card and may be open to suspicion. On the other hand a corresponding line was also observed on a tincalconite pattern made with Fe radiation and there are no good grounds for attributing it to impurity.

The indexing of the pattern clearly shows that the lattice is rhombohedral. No systematic extinctions indicating glide planes are noted. Taking account of etch figures on $\{10\bar{1}1\}$ indicating absence of symmetry planes Minder concluded that the possible space groups are $C_{2v}^2 - R\bar{3}$ and $D_{3h}^7 - R32$. He excluded $C_3^4 - R3$ on morphological grounds and gave preference to group $R\bar{3}$. Observations here reported are not at variance

TABLE 3. POWDER X-RAY DIFFRACTION PATTERN OF TINCALCONITE COMPARED with X-RAY DATA ON ARTIFICIAL $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$

Tincalconite				Artificial $\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$			
Hexagonal indices <i>hkil</i>	Rhombohedral indices <i>hkl</i>	Calculated spacings*	Powder pattern Cu K_α radiation spacing intensity		Minder† Cu rot. pattern intensities	ASTM card 2233† <i>d</i> <i>I</i>	
10 $\bar{1}$ 1	100	8.815	8.83	7	st.st.		
2 $\bar{1}$ 10	1 $\bar{1}$ 0	5.599	5.60	5	m.st.	5.6	0.13
0221	11 $\bar{1}$	4.736	4.73	5		4.72	0.13
20 $\bar{2}$ 2	200	4.407	4.42	9	st.st.st.	4.40	0.60
01 $\bar{1}$ 5	221	3.874	3.93	6 (broad)			
3 $\bar{1}$ 21	2 $\bar{1}$ 0	3.611	3.61	3	m.st.		
1232	21 $\bar{1}$	3.463	3.47	7		3.44	0.53
3030	2 $\bar{1}$ $\bar{1}$	3.233	3.22	2			
2134	310	3.011	3.03	2	st.§	2.99	0.20
1126	321	2.974					
3033	300	2.938	2.94	10	st.st.	2.94	1.00
0333	22 $\bar{1}$	2.938					
2240	20 $\bar{2}$	2.799	2.79	5		2.76	0.13
4132	3 $\bar{1}$ 0	2.606	2.60	6 (broad)	m.	2.60	0.33
2243	31 $\bar{1}$	2.601					
4041	3 $\bar{1}$ $\bar{1}$	2.408	2.412	4		2.40	0.06
0009	333	2.347	2.350	2			
3145	410	2.269	2.278	2	st.	2.26	0.13
4044	400	2.203	2.205	8	st.st.	2.19	0.67
2246	420	2.194					
4150	312	2.116	2.112	1			
1347	430	2.008	2.022	7	st.st.st.	2.01	0.33
022·10	442	1.950	1.954	5		1.94	0.27
5052	41 $\bar{1}$	1.908	1.912	2			
6330	330	1.866	1.866	4	m.st.	1.84	0.13
4156	510	1.814					
6242	420	1.806					
3363	412	1.804	1.811	7+	st.	1.80	0.40
2249	531	1.799					
3257	520	1.791					
5055	500	1.763	1.771	3	m.	1.65	0.07
			1.662	4			
			1.618	2—			
plus about 30 more lines.							

* For planes in rhombohedral cell, $a_r=9.56$, $\angle\alpha=71^\circ42'$, found by Minder, *op. cit.*, p. 304

† Card marked "H" to indicate that data are from Hanawalt of Dow Chemical Co.

‡ For planes in the zone [001] only. As observed on a Cu rotation pattern [001], Minder's Tabelle IV, *op. cit.*, p. 305.

§ Misprint in Minder's Tabelle IV, 2133 for 2134.

with these conclusions but there is some doubt whether the scant morphological data justify any preference among the three space groups permitted by the x -ray and etch figure evidence.

ASSOCIATED MINERALS

The core in which the tincalconite crystals were found has a layered structure so that one might speak of a "stratigraphy" of the core. Unfortunately there is no evidence to indicate what is top or bottom of the core and so the sequence of the layers remains in doubt. Three layers are readily discernible, each making up about one third of the total thickness of 6 or 7 centimeters but the layers vary somewhat in thickness, possibly due to local compaction of the initially very porous material. The "stratigraphy" of the core is essentially:

- A. Whitened prismatic borax, up to 5 mm. long, much pore space, a very few grayish tincalconite crystals
- B. Fine-grained, white, salts in which are embedded numerous clear tincalconite crystals
- C. Reticulated mass of lath-like trona crystals.

Layer A is partly banded due to crystal size variation, but this is not persistent laterally. The layer consists almost wholly of whitened borax. This is in fact tincalconite pseudomorphous after borax as mentioned above. Microscopic examination shows that the pseudomorphing tincalconite is fairly coarse, up to 0.2 mm. or more in dimensions. All the larger fragments show spindly voids arranged at 60° to each other in (0001). In one finer band of borax there are a few grayish equidimensional tincalconite crystals up to 1 mm. in diameter. This tincalconite has copious inclusions of northupite, up to 0.05 mm., and gaylussite, up to 0.01 mm., certainly identifiable by crystal form and optical properties. A trace of trona adheres to the surface.

The change from layer A to layer B is rather abrupt, the whitened borax at the boundary being somewhat finer. The dense white to yellowish salt of layer B encloses numerous clear crystals and clusters of tincalconite which reach up to 4 or 5 millimeters in maximum dimension and were described above. The clear euhedral tincalconite crystals always enclose sharp octahedrons of northupite up to 0.06 mm. from tip to tip and some oval spaces, voids, bubbles or enclosed brine. It was found that each tincalconite crystal of average size (2 mms.) encloses several score northupite crystals. Even so the contamination is only a fraction of one per cent.

The groundmass of layer B consists largely of fine grained tincalconite with embedded laths of trona. There is often a bit of open space about the large tincalconite crystals. By increase in size and abundance of trona this layer grades into layer C which consists almost wholly of reticulated

trona laths, up to 3 mms. or more in length, with a good deal of open space. The trona is very clean and vitreous and rather uniform in habit with the forms {100}, {111} and {001}, flattened parallel to the front pinacoid and elongated along the *b* axis.

All of the minerals found with the tincalconite are well known in the Searles Lake deposits. They constitute only a small part of the full assemblage of minerals known from this locality. A list of the minerals in the Searles Lake saline deposits is given in Table 4. This is an amplifica-

TABLE 4. MINERALS OF THE SEARLES LAKE SALINE DEPOSITS

Discovered at Searles Lake		Year of discovery	Common in oceanic salt deposits	Common in other lacustrine salt deposits
Hanksite	$9\text{Na}_2\text{SO}_4 \cdot 2\text{Na}_2\text{CO}_3 \cdot \text{KCl}$	1885	Calcite	Trona
Sulphohalite	$2\text{Na}_2\text{SO}_4 \cdot \text{NaCl} \cdot \text{NaF}$	1888	Gypsum	Nahcolite
Northupite	$\text{MgCO}_3 \cdot \text{Na}_2\text{CO}_3 \cdot \text{NaCl}$	1896	Anhydrite	Natron
Pirssonite	$\text{CaCO}_3 \cdot \text{Na}_2\text{CO}_3 \cdot 2\text{H}_2\text{O}$	1896	Halite	Gaylussite
Tychite	$2\text{MgCO}_3 \cdot 2\text{Na}_2\text{CO}_3 \cdot \text{Na}_2\text{SO}_4$	1905	Glauberite	Thenardite
Searlesite	$\text{NaB}(\text{SiO}_3)_2 \cdot \text{H}_2\text{O}$	1914	Glaserite	Mirabilite
Schairerite	$\text{Na}_2\text{SO}_4 \cdot \text{Na}(\text{F}, \text{Cl})$	1931		Ulexite*
Burkeite	$2\text{Na}_2\text{SO}_4 \cdot \text{Na}_2\text{CO}_3$	1935		Borax
				Tincalconite

* Found at Searles Lake according to Dr. Joseph Murdoch. (Letter to A. P. dated February 5, 1948.)

tion and rearrangement of a list published by H. S. Gale,¹² minerals found since 1915 being added and the minerals grouped to show the mineralogical peculiarities of the Searles Lake assemblage and to suggest its relations with other salt lake deposits and with oceanic salt deposits.

FORMATION OF THE TINCALCONITE

The mode of occurrence suggests that the tincalconite crystals formed directly from solution. The hole from which the core was taken is filled with brine. The composition of this brine varies only slightly with depth and is very similar to the brines whose composition was reported by H. S. Gale.¹³ A sample from a depth of 115 feet has a specific gravity of 1.304, temperature 74° F. (23.3° C.) and the following composition:¹⁴

¹² *Loc. cit.*, p. 297. Besides these minerals quartz, sulfur, realgar, and a few other sulfides have been found with the Searles Lake salts. The brines also show a sulfide content reported as Na₂S.

¹³ *Op. cit.*, p. 276.

¹⁴ Letter from J. Ryan to D. L. Sawyer, dated 11-29-47.

KCl	3.04%	Na ₂ SO ₄	5.87%
Na ₂ B ₄ O ₇	1.96	NaCl	16.32
Na ₂ CO ₃	6.60	Na ₂ S	0.35

Unfortunately it is not possible to state from experimental data with which crystalline phases such a solution would be in equilibrium.

If a solution of sodium tetraborate is crystallized at room temperature it yields the decahydrate. The pentahydrate is formed by crystallization from the pure aqueous solution only above 61° C. From solutions containing other ions the pentahydrate forms at lower temperatures.¹⁵ If sulfate ion is also present the pentahydrate forms down to 49.3° C., at that temperature being in equilibrium with solution together with the decahydrate and Na₂SO₄ (thenardite). If both sulfate and ammonium ions are present it forms down to 41.7° C., at that temperature being in equilibrium with solution together with the decahydrate, Na₂SO₄ and (NH₄)₂B₄O₇·4H₂O. Presumably in the presence of still other ions Na₂B₄O₇·5H₂O may crystallize directly from solution at still lower temperatures. This may have occurred in the crystallization of tincalconite at Searles Lake.

¹⁵ International Critical Tables, vol. IV, pages 239, 346 and 331, New York (1928).

GOLD CRYSTALS FROM THE SOUTHERN APPALACHIANS

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ABSTRACT

Euhedral gold crystals are extremely rare compared with the amount of the metal produced, and very few have been preserved. Well-formed rhombic dodecahedra are reported for the first time from the Southern Appalachians. The crystals were obtained from placer deposits in Greenville County, South Carolina. Poorly-developed crystals, dendritic forms and wire gold have been reported from Georgia.

Euhedral crystals and filiform gold develop in open spaces, for gold is too soft and malleable to displace most other minerals. Also because of these properties its crystalline structure is readily destroyed by impact or even by polishing, but the high mobility of the atoms makes recrystallization easy. Euhedral crystals are deposited from solutions occupying cavities. Wire-like forms of the native metals result when the material for growth is available in only one direction, the wires being pushed out into the cavities by the addition of atoms at their base from solutions occupying small pore spaces in the walls.

INTRODUCTION

Gold in well-formed crystals is extremely rare compared with the amount of the metal produced. The few euhedral crystals preserved in mineral collections have been recovered mostly from placer deposits, though the softness and malleability of gold make destruction of form during transportation highly probable. Primitive methods of recovering gold from such deposits are more favorable for the discovery of crystals than the methods now generally employed in placer and lode mines. Also the intrinsic value of gold tends to prevent preservation of crystals that would be of scientific value.

Gold has been mined in the Southern Appalachians from placer deposits and veins for more than 150 years, and over 1,622,000 troy ounces (50,500 Kg.) of the metal have been produced. Therefore, it is rather remarkable that euhedral crystals of gold have not been reported previously from this region, but the writer has found no reference to such crystals in the literature.

Genth (1859, p. 254-255) reported gold from Spottsylvania County, Va., "showing very distinctly one rhombohedron, scalenohedron and basal plane," but he states that "it is a coating on tetradymite and evidently a pseudomorph after it." Gold in irregular crystalline masses, wires and dendritic forms has been found at the Loud mine in White County, Ga. (Blake, 1885, p. 584, and Jones, 1909, pp. 45, 207, 208 and Pl. II, fig. 1.), but well formed crystals have not been reported.

EUHEDRAL GOLD CRYSTALS FROM GREENVILLE COUNTY, S. C.

In 1938 the writer found euhedral crystals in a sample of gold presented to him by Mr. S. N. Boozer who had recovered it by panning stream gravels in the northeastern part of Greenville County, S. C. The total weight of the sample is 1.3817 gms. In size the gold ranges from a small nugget of 0.1 gms. to fine dust-like particles. Most of the gold is angular to subangular, but the larger pieces are smoothed and flattened. Some pieces are cavernous, as though the gold had been deposited as an interstitial filling, partly surrounding older minerals. Rectangular cavities suggest deposition about pyrite crystals. Mineral inclusions are present and one piece of gold is magnetic because of included magnetite or ilmenite.

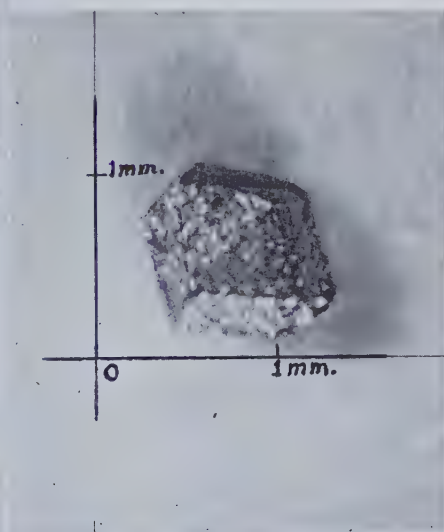


FIG. 1



FIG. 2

FIG. 1. Gold Crystal (Rhombic Dodecahedron) from Greenville County, S. C.

FIG. 2. Gold Crystal with Cavity from Greenville County, S. C.

One well-formed crystal (Fig. 1), about 0.8 mm. in diameter and weighing 0.0039 gm., is a simple rhombic dodecahedron slightly flattened parallel to two faces and showing no evidence of modifying faces. The crystal faces are slightly pitted and the edges rounded by abrasion during transportation, but the luster is high. Some of the edges look as though they may have been slightly raised above the faces.

A larger dodecahedron (Fig. 2), about 1.7 mm. in diameter and 0.0324

gm. in weight, is more scarred and rounded by abrasion, but it likewise shows no evidence of combinations with other isometric forms. This crystal contains a small but relatively deep cavity.

Two small spheroidal grains present in the collection possibly represent well-rounded dodecahedra. An irregular shaped particle of gold shows a single, smooth, triangular face, and another shows a small square face, both having a high luster. These possibly represent the faces of other forms such as the octahedron and cube, but no conclusive evidence was found of the presence of any crystal form other than the simple rhombic dodecahedron.

The country rock in the area from which the gold crystals were obtained consists chiefly of gneisses and schists with intrusions of granite and diabase. The gold occurs in small quartz stringers cutting sericitic schists and also, with pyrite, as scattered impregnations in the schist. The area is in the upper Piedmont where deep weathering is prevalent under divides. These conditions are favorable for the concentration of gold in stream beds, and, according to Sloan (1908, p. 32), limited placer deposits along a few streams in the area were worked successfully during the early days of mining, but efforts to recover gold from the veins were not encouraging. The appearance of the gold examined suggests that some of the particles have traveled farther than others but that most of it, including the euhedral crystals, has been transported for only a very short distance.

GOLD CRYSTALS FROM GEORGIA

Beautiful specimens of native gold from the Loud mine in White County, Ga., are preserved in the Museum of the Georgia Geological Survey, where they were examined recently by the writer. Part of the gold is in irregular masses or nuggets showing no evidence of crystal form, but the collection contains much gold in the form of strings of closely-crowded, small crystals with parallel orientation. Branching, resulting in dendritic, arborescent and reticulated forms, is common. This type of crystallization is closely similar to that of gold from the White Bull mine in Oregon, described by Dana (1886, 132-138).

Dana explains that the faces of the tiny "rhombohedrons" forming the strings are those of a tetragonal trisoctahedron elongated in the direction of the trigonal axis with suppression of other faces. Repeated twinning on {111}, together with branching at 60° results in the dendritic and reticulated forms.

One specimen of gold quartz, probably the one shown by Jones (1909, Pl. II, fig. 1), contains a group of rounded grains or crystals, each 3 to 4 mm. in diameter, but the faces are so imperfect that the crystal form

could not be determined with any degree of certainty. Cavernous faces seem to be present, but no smooth planes, as all edges are much rounded.

No true wire gold is present in the collection.

CRYSTAL FORMS AND STRUCTURE

According to Dana's *System of Mineralogy* (Palache, Berman and Frondel, 1944, Vol. 1, p. 90-95) gold crystallizes in the hexoctahedral class of the isometric system, the commonest forms being the octahedron {111}, rhombic dodecahedron {011}, cube {001}, and tetragonal trisoctahedron or trapezohedron {113}. The tetrahexahedron {012} and hexoctahedron {124} are also reported; and several other forms, such as {014}, {013}, {025}, {118}, {114}, {112}, {223}, {3·5·11}, {237}, {123} and {345}, are rare or uncertain.

Shepard (1851, p. 231-232) describes a crystal of native gold from California having the shape of a pentagonal dodecahedron. He states that it is $\frac{2}{3}$ inch in diameter, and "presents distinct traces of the raised edges so characteristic of the native gold crystals of California, and which militates against the idea that these forms were produced in moulds left vacant from the decomposition of iron pyrites." This suggests that gold has the lower symmetry of the diploidal class, but gold crystals with cubic faces showing the characteristic striations of this class have not been reported.

Evidence from many crystals and from x-ray analyses shows conclusively that gold, and all the other metals of the gold group, crystallize in the hexoctahedral division and have a similar structure, the atoms being arranged in the simple face-centered cubic lattice with 4 atoms to the unit cell. If Shepard's crystal is not a pseudomorph it may be structurally a tetrahexahedron with half of its faces missing.

Most gold crystals reported are not simple forms but combinations of two or more forms, and some are highly complex. Simple cubes are very rare, though cubes combined with other forms, especially the octahedron, are relatively common. The octahedron is probably the commonest simple form, and is said by Blake (1885, p. 576-577) to be the characteristic form in California, though most of the crystals are flattened parallel to {111}, or otherwise distorted.

The rhombic dodecahedron seems to be more abundant in Australia than elsewhere. Professor Booth of the Philadelphia mint in a letter to Dana (1869, p. 8) states "that one lot of Australian gold worth about \$4,000, submitted to him in 1853, consisted of grains from the size of a large pea to small sand, all of which were more or less perfect dodecahedrons." However, some of the dodecahedra reported from Australia are not simple crystals of that form. Blake (1885, p. 575) states that "At

Mount Ivor numerous small solid dodecahedrons were obtained retaining portions of the cubic faces, thus forming cubo-octahedrons," and Liveridge (1907, p. 143) shows that apparently simple gold crystals, such as well-formed rhombic dodecahedra from New South Wales, on etching, yield ample evidence that they are not internally homogeneous but are in reality highly complex, and are composed of a number of individuals. These crystals may be pseudomorphs or they may be rhombic dodecahedra that have recrystallized.

The tetrahexahedron, trapezohedron (tetragonal trisoctahedron), trigonal trisoctahedron, and hexoctahedron are rare as simple forms and are not very common in combinations.

Euhedral gold crystals apparently are produced only in cavities. Many minerals have such a strong tendency to develop crystal form that, in growing, they make room for themselves by displacing or by replacing most other minerals. Gold is so soft and malleable that it has little tendency to displace other minerals, and other minerals growing concurrently or subsequently may easily exert sufficient pressure to destroy the form of gold crystals. Gold crystals may grow around and thus enclose other minerals, and, in the same way, other minerals enclose gold crystals.

DISTORTED AND FILIFORM CRYSTALS

Gold crystals, and especially octahedra, commonly show recessed or cavernous faces and salient edges, a characteristic usually associated with relatively rapid growth and therefore, insufficient time for molecules to diffuse to the interior of faces. The dendritic, arborescent and reticulated forms are also commonly associated with rapid growth. Gold is deposited from solutions containing very small amounts of the metal as compared with the other vein-forming minerals present, but it is very easily precipitated from solution. Therefore a dilute gold solution may be supersaturated with respect to gold crystals.

Some writers have referred to the thread-like arborescent forms, made up of small crystals closely crowded upon each other, as "wire gold," but it would seem preferable to reserve this term for the smooth-surfaced wire-like forms of the metal. Silver occurs in this form much more commonly than gold. In some specimens several small wires gradually merge to form a single larger wire. They do not branch off at an abrupt angle as most of the dendritic or arborescent specimens. The wires are commonly curved and in some cases form a tangled mass of fine wires that more or less fill small cavities. The resemblance of the filiform varieties of the metals to the hair-like crystals of epsomite, chalcantite and alunite, that grow out from the walls of caves and that have been produced experimentally by the writer, suggest that the metallic wires have been formed in a similar manner. According to this hypothesis the wires are

pushed out into cavities through the addition of molecules at their base from solutions occupying small pore spaces in the wall. The wire-like form is due to the fact that material for growth was available only at their base. If the depositing solutions were in contact with the wires elsewhere some crystal faces should be formed.

RECRYSTALLIZATION OF GOLD

Because of its softness and malleability, the crystalline structure of gold is easily destroyed. Beating of pure gold foil makes the metal harder and less plastic, and Beilby (1905, p. 223) has shown that this condition is accompanied by the disappearance of all outward traces of crystalline structure. The complete destruction of the crystalline units takes place only at the surface. "By carefully etching the surface in stages by means of chlorine water or aqua regia the successive layers below the surface were disclosed. The surface itself was vitreous. Beneath this was a layer of minute granules, and lower still the distorted and broken-up remains of crystalline lamellae and grains were embedded in a vitreous and granular matrix. The vitreous-looking surface layer represents the final stage in the passage from soft to hard, from crystalline to amorphous. By heating the beaten foil its softness was restored, and, on etching the annealed metal, it was found that the crystalline structure also was fully restored." Beilby (1905, p. 222) states that "during polishing the disturbed surface film behaves exactly like a liquid under the influence of surface tension."

Bragg (1914, p. 355-360) found that the outer surface of copper crystals, which have been much battered and distorted, retains little of the regular crystalline arrangement. Attempts to grind crystal faces artificially also destroyed the crystalline character of the surface and so prevented the reflection of x -rays from the face. Faces were therefore prepared for x -ray spectrographic work by dissolving the surface with acid.

The recrystallization of amorphous gold that occurs quickly during annealing probably takes place slowly at relatively low temperatures, for gold atoms possess a high degree of mobility. Roberts-Austen (1896, p. 284-285), found that solid diffusion of gold into lead was relatively rapid at a temperature of 251° C. (70 mm. in 31 days), and that the rate of diffusion is still readily measurable at 100° C. Slow recrystallization following distortion and battering probably explains the rhombic dodecahedra composed of a number of individual crystals that were reported by Liversidge.

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X-RAY MEASUREMENTS ON BRACKEBUSCHITE AND HEMATOLITE

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ABSTRACT

New observations on brackebuschite from Sierra de Cordoba, Argentina, and on hematolite from the Moss mine, Nordmark, Sweden, combined with existing chemical analyses yield the following descriptions of these minerals.

Brackebuschite: monoclinic, probable space group $P2_1/m$; the unit cell with $a=8.92$, $b=6.16$, $c=7.69$ Å, $\beta=111^\circ 47'$; contains $2[\text{Pb}_2(\text{Mn,Fe})(\text{VO}_4)_2 \cdot \text{H}_2\text{O}]$. Specific gravity 6.05 (measured), 6.07 (calculated).

Hematolite: hexagonal $-R$; probable space group $R\bar{3}$; the unit cell with $a=8.27$, $c=36.51$, $r=13.07$ Å, $\alpha=36.53'$, $a:c=1:4.415$; contains $\text{Mn}_{10}\text{Mg}_2\text{Al}_3(\text{AsO}_4)_3(\text{OH})_{24}$. Specific gravity 3.49 (measured), 3.48 (calculated). The observed crystal forms are $c(0001)$ and the negative I order rhombohedra $Q(0.2.2.13)$, $P(01\bar{1}5)$ and $S(01\bar{1}2)$.

Brackebuschite and hematolite are two rare minerals, encountered by the authors of volume II of Dana's *System*, on which x -ray observations have not been made. The opportunity to examine these two minerals was provided by typical specimens kindly loaned from Harvard Museum (HM) by Dr. C. Frondel. An additional specimen of each mineral was later borrowed from the United States National Museum (USNM) through the kindness of Mr. E. P. Henderson.

BRACKEBUSCHITE

Brackebuschite, a hydrous vanadate of lead, manganese and iron, occurs at several localities in Sierra de Cordoba, Argentina. On the specimens at hand (HM 96255, USNM C4195), both from the Venus Mine, the mineral occurs in small flattened prismatic crystals, rhombic in cross section and striated parallel to the elongation. The crystals are dark brown to black and transmit red light on thin edges. Excellent x -ray rotation and Weissenberg films were obtained by turning a crystal first about the axis of elongation and later about an axis perpendicular to the elongation. These films indicate monoclinic symmetry with elongation along the 2-fold symmetry axis and yield the cell dimensions:

$$a=8.92, b=6.16, c=7.69 \text{ Å}; \beta=111^\circ 47'.$$

The systematically missing spectra conform to the conditions: (hkl) present in all orders, $(0k0)$ present only with k even, $(h0l)$ present in all orders. The space group is therefore $P2_1/m$ if the crystals are holohedral. The crystals show the pinacoids $a(100)$ and $c(001)$ in almost equal development. No terminal faces were observed. The measured angle $c \wedge a$ agrees roughly with the β angle determined from x -ray films.

The only available analyses were made by Doering, quoted by Ram-melsberg (1880) and again by Dana (1892). The first of the three closely agreeing analyses (1 of Table 1), when combined with the cell volume and measured specific gravity 6.05 (Berman balance) give the empirical cell contents (2) and atomic contents (3). These numbers clearly indicate the ideal structural formula $\text{Pb}_4\text{MnFe}(\text{VO}_4)_4 \cdot 2\text{H}_2\text{O} = 2[\text{Pb}_2(\text{Mn,Fe})(\text{VO}_4)_2 \cdot \text{H}_2\text{O}]$ with the numbers of atoms (4) and the calculated composition (5). The specific gravity calculated for the ideal cell content is 6.07, in close agreement with the measured value.

TABLE 1. BRACKEBUSCHITE: ANALYSIS AND CELL CONTENT

	1	2	3		4	5	
PbO	61.00	3.88	Pb	3.88	4	PbO	62.20
FeO	4.65	.92	Fe	.92	1	FeO	5.01
MnO	4.77	.95	Mn	.95	1	MnO	4.94
ZnO	1.29	.22	Zn	.22			
CuO	0.42	.07	Cu	.07			
V ₂ O ₅	25.32	1.97	V	3.94	4	V ₂ O ₅	25.34
P ₂ O ₅	0.18	.02	P	.04			
H ₂ O	2.03	1.60	H	3.20	4	H ₂ O	2.51
			O	17.59	18		
	99.66						100.00

The following table gives the *x*-ray powder pattern of brackebuschite for $\text{CuK}\alpha$ radiation, indexed as far as $\vartheta = 26.6^\circ$.

The close chemical similarity has suggested to some authors that brackebuschite and pyrobelonite may be members of a series. The crystallographic data presented here fail to show any relationship to pyrobelonite which is orthorhombic with a probable structural formula $4[(\text{Mn,Pb})_2\text{VO}_4(\text{OH})]$ (Richmond, 1940). The structural formula of brackebuschite clearly suggests that the mineral belongs to the chemical type $\text{A}_3(\text{XO}_4)_2 \cdot n\text{H}_2\text{O}$. In the classification of minerals of this type given by Wolfe (1940) brackebuschite would belong to a family $\text{A}_3(\text{XO}_4)_2 \cdot \text{H}_2\text{O}$, not recognized by Wolfe. No other mineral representatives of this family have yet been noted.

HEMATOLITE

Hematolite, a basic arsenate of manganese and aluminum, occurs at the Moss Mine, Nordmark, Vermland, Sweden. On the specimens (HM, Boston Society of Natural History, Brigham coll. 396 and USNM C4228) available for this study from the type locality, the mineral oc-

TABLE 2. BRACKEBUSCHITE: $\text{Pb}_2(\text{Mn, Fe})(\text{VO}_4)_2 \cdot \text{H}_2\text{O}$
 Monoclinic, $P2_1/m$; $a=8.92$, $b=6.16$, $c=7.69$ Å; $\beta=111^\circ 47'$; $Z=2$

<i>I</i>	$\vartheta(\text{Cu})$	<i>d</i> (meas.)	(<i>hkl</i>)	<i>d</i> (calc.)	<i>I</i>	$\vartheta(\text{Cu})$	<i>d</i> (meas.)	(<i>hkl</i>)	<i>d</i> (calc.)
1	5.35	8.26 Å	(100)	8.283 Å	2	19.8	2.27 Å	{ $(\bar{2}22)$ $(\bar{3}03)$ }	{2.282 Å 2.266}
8	8.95	4.95	(110)	4.943					
2	9.6	4.62	(101)	4.625	4	21.2	2.13	($\bar{3}21$)	2.139
$\frac{1}{2}$	10.8	4.11	(200)	4.141					
2	12.1	3.67	(111)	3.699	4	21.8	2.07	{ $(\bar{4}11)$ (400) }	{2.090 2.070}
2	13.1	3.40	(202)	3.399					
10	13.7	3.25	(112)	3.257	$\frac{1}{2}$	22.2	2.04	($\bar{3}22$)	2.042
5	14.5	3.08	{(012) (020)}	{3.089 3.080}					
6	15.0	2.98	{ $(\bar{2}12)$ $(\bar{3}01)$ }	{2.976 2.973}	1	22.9	1.979	{(031) $(\bar{1}31)$ $(\bar{1}23)$ }	{1.973 1.966 1.966}
8	16.2	2.76	{(211) (300)}	{2.780 2.761}	3	23.8	1.909	{(204) $(\bar{1}04)$ }	{1.918 1.900}
1	17.2	2.61	(112)	2.631	3	24.3	1.872	{ $(\bar{4}13)$ (103)}	{1.872 1.859}
2	17.9	2.51	{ $(\bar{2}21)$ $(\bar{3}12)$ }	{2.514 2.495}	3	24.8	1.839	{ $(\bar{3}04)$ (230)}	{1.843 1.840}
1	18.9	2.38	{ $(\bar{1}22)$ (003)}	{2.402 2.380}	3	25.3	1.802	($\bar{4}21$)	1.802
2	19.3	2.33	(022)	2.332	6	26.6	1.720	(213)	1.724

<i>I</i>	$\vartheta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\vartheta(\text{Cu})$	<i>d</i> (meas.)	<i>I</i>	$\vartheta(\text{Cu})$	<i>d</i> (meas.)
3	28.3	1.625	1	34.6	1.356	$\frac{1}{2}$	50.4	1.000
4	30.1	1.536	2	38.4	1.240	$\frac{1}{2}$	51.6	0.982
1	32.1	1.449	$\frac{1}{2}$	45.5	1.080	1	57.1	0.917
2	33.0	1.414	$\frac{1}{2}$	47.8	1.040	$\frac{1}{2}$	59.1	0.898
3	33.9	1.381	$\frac{1}{2}$	49.1	1.019	$\frac{1}{2}$	60.7	0.883

curs as tiny tabular crystals and aggregates in small vugs and fissures in crystalline limestone. The crystals are brownish-red to black in colour and orange to reddish-orange in thin flakes by transmitted light. The perfect basal cleavage shows a pearly lustre.

Sharp rotation and Weissenberg *x*-ray films obtained with FeK radiation by rotating a crystal about *c*, *a* and $[21\bar{3}0]$ indicate an essentially rhombohedral lattice with dimensions:

$$a=8.27, c=36.51; r=13.07 \text{ Å}, \alpha=36^\circ 53'.$$

The systematically missing spectra conform very closely to the conditions ($hki\bar{l}$) present only with $h+i+l=3n$, characteristic of a rhombohedral lattice. A few weak diffraction spots on the Weissenberg resolution ($h0\bar{h}l$) which do not conform to the above lattice extinction condition indicate a hexagonal superstructure with the dimensions given. The Weissenberg resolution ($hki0$) with diffractions ($30\bar{3}0$), ($11\bar{2}0$), ($22\bar{4}0$) and ($41\bar{5}0$) clearly indicates lack of symmetry about the ($h0\bar{h}0$) rows. On x -ray precession camera films about c with $\text{MoK}\alpha$ and $\text{CuK}\alpha$ radiation, the zero level ($hki0$) shows 6-fold symmetry without planes of symmetry; the levels ($hki1$, 2, 3, 4 and 5) each show 3-fold symmetry without planes of symmetry. Therefore the Laue class symmetry is clearly $\bar{3}$ and the probable space group is $R\bar{3}$.

The geometrical ratio of Sjögren (1885) is related to the structural elements if the c length is multiplied by 5, giving the comparison:

$$a:5c = 1:4.4425 \text{ (Sjögren, goniometric)}$$

$$a:c = 1:4.415 \text{ (Berry, } x\text{-ray).}$$

The crystals are thick tabular with a triangular basal pinacoid bevelled by I order rhombohedron faces which are striated horizontally. Two circle goniometer ρ angles obtained from 6 crystals are recorded below in comparison with Sjögren's measured and calculated angles and angles calculated from the structural elements. X -ray precession camera films about c on one of the measured crystals clearly indicate that the crystal forms, other than $c(0001)$ are properly negative I order rhombohedra.

TABLE 3. HEMATOLITE: MEASURED AND CALCULATED ρ ANGLES

Sjögren			Berry		
	measured	calc.		measured	calc.
—	average	—	(0.1.1.29)	range	average
q ($30\bar{3}4$)	—	$37^{\circ}34\frac{1}{2}'$	Q ($0.2.\bar{2}.13$)	$8^{\circ}01' - 12^{\circ}18'$ (4)	$10^{\circ}04'$
r ($10\bar{1}1$)	$45^{\circ}44'$	$45^{\circ}44'$	P ($01\bar{1}5$)	—	—
s ($20\bar{2}1$)	64 53	64 01	($02\bar{2}5$)	43 55 - 50 15 (15)	45 54
—	—	68 42	S ($01\bar{1}2$)	—	—
				64 59 - 70 48 (16)	67 37
					63 53
					68 35

In view of the poor quality of the reflections obtained from the measured crystals the agreement between measured and calculated angles is fair. Sjögren's elements, transformed to the structural setting, appear to fit the measured angles best. The comparison of angles indicates that Sjögren's forms $s(2021)$ and $q(30\bar{3}4)$ are probably equivalent to $S(01\bar{1}2)$

TABLE 4. HEMATOLITE: ANALYSES AND CELL CONTENT

	1	2	3	4	5		6	7		8
MnO	46.86	50.98	10.33	10.67	10.50	Mn	10.50	10	MnO	47.17
MgO	6.66	5.38	2.58	1.98	2.28	Mg	2.28	2	MgO	5.36
CaO	0.66	0.71	0.18	0.19	0.19	Ca	0.19	—	—	—
Al ₂ O ₃	6.39	8.61	0.98	1.25	1.19	Al	2.38	3	Al ₂ O ₃	10.17
Fe ₂ O ₃	1.01		0.15							
As ₂ O ₅	21.55	22.54	1.47	1.46	1.47	As	2.94	3	As ₂ O ₅	22.93
H ₂ O	13.93	14.02	12.09	11.55	11.87	H	23.74	24	H ₂ O	14.37
Insol.	0.64					O	35.76	36		
	97.70	102.24								100.00

and $Q(0.2\bar{2}.13)$, respectively, in the structural setting. Likewise the form $t(70\bar{7}3)$, observed by Lorenzen (1884) is equivalent to $S(01\bar{1}2)$. The new form $(0.1\bar{1}.29)$ recorded here, was observed on two crystals as a very narrow face bevelling the edge of $c(0001)$.

TABLE 5. HEMATOLITE: $(\text{Mn,Mg})_4\text{AlAsO}_4(\text{OH})_8$
Hexagonal, $R\bar{3}$; $a=8.27$, $c=36.51$; $r=13.07\text{\AA}$; $\alpha=36^\circ 53'$, $Z(r)=3$

<i>I</i>	$\vartheta(\text{Fe})$	<i>d</i> (meas.)	(<i>hkl</i>)	<i>d</i> (calc.)	<i>I</i>	$\vartheta(\text{Fe})$	<i>d</i> (meas.)	(<i>hkl</i>)	<i>d</i> (calc.)
8	9.1°	6.12Å	(0006)	6.085Å	10	23.9	2.39Å	$\begin{Bmatrix} (21\bar{3}7) \\ (30\bar{3}0) \end{Bmatrix}$	$\begin{Bmatrix} 2.403\text{\AA} \\ 2.387 \end{Bmatrix}$
3	9.9	5.63	(10 $\bar{1}$ 4)	5.634					
5	10.7	5.20	(01 $\bar{1}$ 5)	5.114					
8	13.5	4.14	(11 $\bar{2}$ 0)	4.135	3	24.6	2.33	(12 $\bar{3}$ 8)	2.328
2	13.8	4.06	(0009)	4.057	1	26.5	2.17	(2.1.3.10)	2.177
3	14.4	3.89	(11 $\bar{2}$ 3)	3.915					
4	16.5	3.40	(11 $\bar{2}$ 6)	3.420	2	27.6	2.09	$\begin{Bmatrix} (1.1.\bar{2}.15) \\ (1.2.\bar{3}.11) \end{Bmatrix}$	$\begin{Bmatrix} 2.098 \\ 2.098 \end{Bmatrix}$
4	17.4	3.22	$\begin{Bmatrix} (1.0.\bar{1}.10) \\ (20\bar{2}5) \end{Bmatrix}$	$\begin{Bmatrix} 3.253 \\ 3.215 \end{Bmatrix}$					
2	18.6	3.03	(0.0.0.12)	3.042	2	29.8	1.948	$\begin{Bmatrix} (2.1.\bar{3}.13) \\ (\bar{1}\bar{3}44) \end{Bmatrix}$	$\begin{Bmatrix} 1.949 \\ 1.941 \end{Bmatrix}$
3	19.0	2.97	(02 $\bar{2}$ 7)	2.952					
1	19.6	2.89	(11 $\bar{2}$ 9)	2.896	4	31.0	1.880	$\begin{Bmatrix} (3.0.\bar{3}.12) \\ (1.2.\bar{3}.14) \end{Bmatrix}$	$\begin{Bmatrix} 1.878 \\ 1.878 \end{Bmatrix}$
3	20.1	2.82	(20 $\bar{2}$ 8)	2.817					
1	21.0	2.70	(21 $\bar{3}$ 1)	2.700	4	33.7	1.745	(2.1.3.16)	1.745
4	21.9	2.60	(21 $\bar{3}$ 4)	2.595	1	37.2	1.601	(4.0.4.10)	1.607
					9	38.3	1.562	(41 $\bar{5}$ 0)	1.563
1	23.4	2.44	$\begin{Bmatrix} (0.0.0.15) \\ (2.0.\bar{2}.11) \end{Bmatrix}$	$\begin{Bmatrix} 2.434 \\ 2.434 \end{Bmatrix}$	2	39.8	1.512	(0.2.2.22)	1.506
					1	45.0	1.369		
					1	47.8	1.307		
					1	52.6	1.218		
					1	57.9	1.143		

The two available analyses of hematolite by C. H. Lundstrom (1 of Table 4) and A. Sjögren (2) (Sjögren, 1885) when combined with the rhombohedral cell volume and the measured specific gravity, 3.49, give the cell contents (3) and (4) and the average cell content (5). The average atomic content of the rhombohedral cell (6) indicates the ideal structural formula $\text{Mn}_{10}\text{Mg}_2\text{Al}_3(\text{AsO}_4)_3(\text{OH})_{24}$ with the numbers of atoms in column (7). The total number of metal atoms is undoubtedly 15 but the numbers of each are uncertain. The formula above has been written with 12 divalent and 3 trivalent metals. The latter, shown as all Al in the formula probably includes both Fe''' and Mn''' . For this reason the calculated specific gravity, 3.48, for this structural formula, is low although

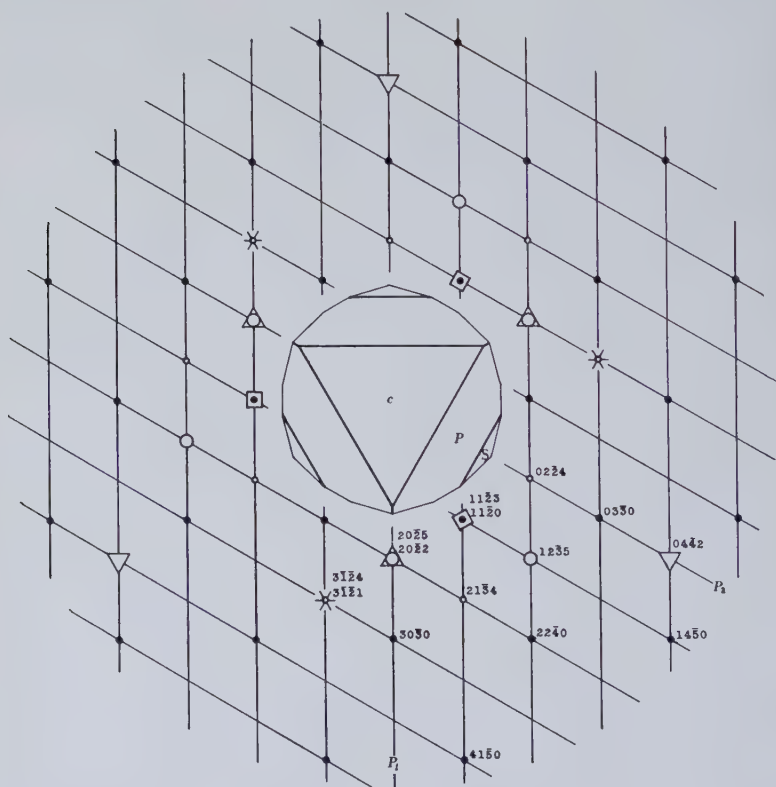


FIG. 1. Hematolite, Nordmark, Sweden: Idealized plan drawing of a typical crystal with $c(0001)$, $P(01\bar{1}5)$ and $S(01\bar{1}2)$ superimposed on the reciprocal lattice projection of diffractions observed on x -ray precession camera films about c (observed diffractions shown as follows: $(hki0)$ as solid circles, $(hki1)$ as small open circle with six rayed star, $(hki2)$ as triangles, $(hki3)$ as squares, $(hki4)$ as small open circles and $(hki5)$ as large open circles. $(41\bar{5}0)$ and $(14\bar{5}0)$ diffractions appear with distinctly different intensities.

it agrees well with the measured value; and the calculated composition differs somewhat from the analyses, principally in the percentage of alumina.

The x -ray powder pattern of hematolite for $\text{FeK}\alpha$ radiation, indexed as far as $\vartheta = 39.8^\circ$, is given in Table 5.

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EFFECTS OF ROTATION OF OBJECTIVES ON THE OPTICAL PROPERTIES OF OPAQUE MINERALS IN POLARIZED LIGHT

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ABSTRACT

It has been found that rotation of the objective causes a change in the configuration of opaque mineral figures, and, in some cases, in their color patterns. Analogous changes are produced in the rotation colors and reflected pleochroism of anisotropic opaque minerals. The changes in the optical figures are apparently governed to an extent by the type of figure viewed. It is suggested that this phenomenon is due to lack of rotational symmetry in the objective.

INTRODUCTION

M. N. Short (5) states in his bulletin on the microscopic determination of the ore minerals that he was unable to duplicate the rotation colors (1) (polarization colors) listed by Van der Veen and Farnham. He points out that rotation colors vary with the adjustment of the nicols, the source of light (arc or incandescent), the intensity of illumination, and the perfection of polish, in addition to the effects produced by the mineral itself.

There is yet another, hitherto unknown, factor, which effectively makes it nearly impossible for different workers to obtain accurately comparable results in the determination of rotation colors. In a continuing investigation of the optical figures obtained with the reflecting microscope (4) (opaque mineral optical figures), the author, at the suggestion of G. T. Hammond (2), Bausch & Lomb Optical Company, investigated the effect of rotation of the objective on the configuration of these optical figures, with some very interesting results.

EXPERIMENTAL PROCEDURE

Bausch & Lomb Optical Company recently lent the author an objective rotating adapter. The adapter consists of two brass rings, with appropriate threading for insertion between the microscope tube and the objective; by using these rings an objective can be rotated about the axis of the microscope. Use of the adapter changes the tube length of the microscope, but has no other effect.

The objective rotating adapter is used by Bausch & Lomb for the express purpose of detecting strain in an objective. The author is indebted to G. T. Hammond (2) for the following description of the procedure employed: "When using the rotating adapter to detect strain in an objective used with transmitted light, we look for a deformation of the interference figure as the objective is rotated. We take a specimen of common type such as mica or quartz and set up the microscope to obtain the normal

position interference figure (centered black cross) of that crystal. Then the objective is rotated and if there is any deformation of the interference figure we know that strain is present in some part of the objective. The degree of deformation of the interference figure indicates the amount of strain which may be present. It is reasonable to assume that the same procedure would work with vertical illumination provided no strain is present in the optical elements between the polarizer and analyzer."

G. T. Hammond suggested that the optical figure obtained from pyrite with an unstrained objective should theoretically be a type I figure rather than the type III figure consistently obtained. Accordingly the rotating adapter was sent on loan for the purpose of determining if the objectives used were strain free. The test was first made on opaque minerals using vertical illumination; totally unsuspected results were obtained. At this time a test with transmitted light had not been made on the objectives in question; on learning of the perplexing results which had been obtained, G. T. Hammond was kind enough to send an objective guaranteed to be strain free.

The objectives used in this investigation, numbered for reference, are No. 1—a Spencer 4 mm. short-mounted objective, No. 2—a B & L 4 mm. short-mounted objective, No. 3—B & L 4 mm. long-mounted objective No. 72140 (manufacturer's serial number), No. 4—B & L 4 mm. long-mounted objective No. BD8659 (on loan from Bausch & Lomb), and No. 5—a Leitz 4 mm. long-mounted objective.

OBSERVATIONS

Using transmitted light and the Bx_a interference figure from a basal section of muscovite, the following results were obtained in the strain test: objective No. 1—a very slight amount of strain in the exact center of the field of view; objective No. 2—same results as for objective No. 1, but the amount of strain is even less; objective No. 3—a barely perceptible and slight amount of strain is evident near the edge of the field of view; objective No. 4—no detectable strain; and objective No. 5—the test was not performed on this objective. It is unfortunate that the tests made in this instance and elsewhere are not complete, but as the instruments on loan have been returned to Bausch & Lomb, this oversight has not been corrected.

The effects of rotation of the adapter with vertical illumination are described below as they were seen on the minerals pyrite, stibnite, and covellite.

A. Pyrite. On a (001) face of pyrite, all objectives so far used in this investigation and elsewhere (16 in number) yield a type III figure, having an "optic angle" of approximately 20° . True, the "optic angle" varies

slightly, but otherwise there is no difference. All the objectives used in this investigation show the same behavior on rotation. As the objective is rotated with the mineral in a stationary position, the isogyres behave in somewhat the same way as does a centered Bx_a interference figure during rotation of the mineral; that is, they come together and they separate into alternate pairs of quadrants four times during rotation of the objective (Fig. 1). Rotation of the mineral, when the objective is

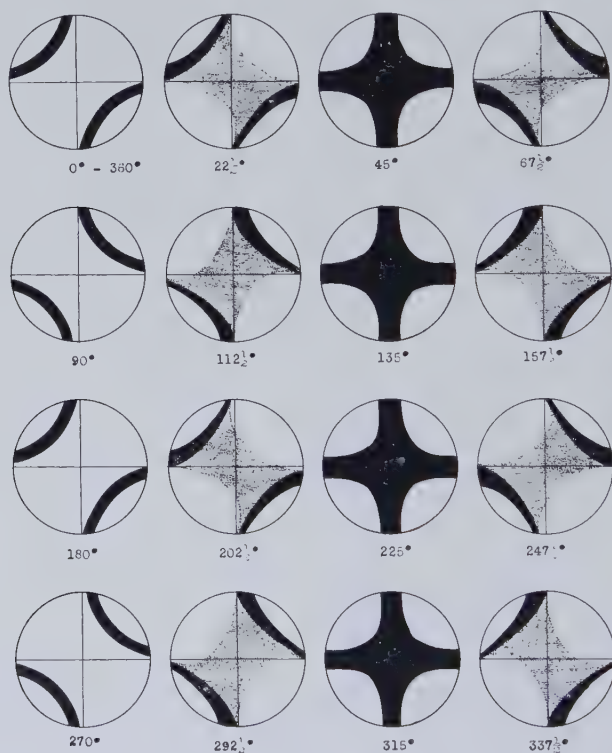


FIG. 1. Effect of rotation of the objective on the type III figure of pyrite (001). The 0° and 180° positions shown above correspond to the type III figure. The gray areas appear only between 0° and 45°.

stationary, does not alter the configuration of the optical figure determined by the position of the objective. It is of interest to note that all objectives show exactly the same configuration without the adapter.

B. Stibnite (010 cl.). Only objectives No. 1 and No. 4 were used in this case. Rotation of objective No. 1 produces the same general effect as on pyrite, but superimposed on the type IV figure of stibnite. The distance between the isogyres, as determined by the orientation of the mineral

with respect to the planes of polarization, is a maximum in each case, which is not exceeded in that orientation, during rotation of the objective; this is also true of covellite. Here again, 16 different objectives, without the adapter, yield the same figure on stibnite (type IV).

With objective No. 4, the results differ slightly from those obtained with objective No. 1. Between the minimum angle position and approximately halfway to the maximum angle position, rotation of the objective causes the same change as does rotation of objective No. 1. Between this position and the maximum angle position, rotation of the objective has little effect, acting only to slightly distort the isogyres.

C. Covellite. (Polished section near (0001)—Subtype II*b*). As with stibnite, only objectives No. 1 and No. 4 were used. Rotation of objective No. 1 causes the same qualitative effect as on pyrite, but superimposed on the subtype II*b* figure; this is true only between the crossed and 45° positions. At the 45° position only the color pattern is affected, roughly in the same sense as when the mineral is rotated with the objective stationary.

The effect on the optical figure of rotating objective No. 4 was masked by myriad internal reflections. Apparently there was little, if any, change in the figure.

With covellite and stibnite, the behavior of the isogyres on rotation of the mineral, with the objective stationary, apparently is the same as that normally seen when the microscope is used without the adapter.

The results obtained with pyrite were communicated to Dr. George Tunell, who suggested the following explanation (3): "Your approach to the problem of the behaviour of the pyrite interference figure by rotating a number of different objectives strikes me as good. Since everything else remained stationary the effect must be due to lack of rotational symmetry of the objectives. The phenomena you are dealing with involve elliptical polarization and it may be that this is one reason why they are so sensitive to lack of rotational symmetry in the objective."

The question then arose; if this were true, would the phenomenon be confined to the opaque mineral optical figures. If not, should not a similar set of results be obtained with those other optical properties of reflecting surfaces, which owe their origin in part to elliptical polarization? Accordingly, the effect of rotating the objective was investigated with respect to rotation colors and reflected pleochroism. As these phenomena are better seen with objectives of lower magnification a Leitz 14 mm. objective was used. The results obtained were checked with objectives of higher magnification. The phenomenon was determined in a qualitative sense only.

A. Rotation Colors. The effects obtained on covellite and stibnite, the two

minerals examined, are similar. The effect of rotation of the objective with the mineral stationary is qualitatively the same as rotation of the mineral with the objective stationary. The actual colors observed with rotation of the objective depends on the orientation of the mineral in relation to the planes of polarization of the microscope, and vice versa. The colors obtained in different positions of either mineral or objective, on rotation of the other are the same as those obtained with different settings of the polarizer; i.e. different angles between the planes of polarization of the polarizer and the analyzer. The same phenomenon was noted with objectives of higher magnification.

B. Reflected Pleochroism. On covellite, rotation of the 14 mm. objective produces no, or a very slight, perceptible change in the reflected pleochroic colors. The changes that take place are best seen with objectives of higher magnification. It appears that changes produced on rotation of the objective are similar to those changes produced in the rotation colors.

SUMMARY

The phenomenon described here presents an interesting and important problem. It has been demonstrated that this phenomenon is not confined to opaque mineral optical figures. This observation lends further support to Tunell's suggestion that the phenomenon is due to lack of rotational symmetry in the objective, accentuated by elliptical polarization. The lack of agreement among different workers in the determination of certain optical features of opaque minerals in polarized light can conceivably find its explanation in this phenomenon. Yet, there is remarkable agreement in these determinations in light of the phenomenon described above.

It seems doubtful that the use of the rotating adapter should cause this phenomenon, as the only way in which it modifies the optics of the equipment used is to increase the tube length of the microscope. Furthermore, the use of the adapter has no effect on interference figures obtained with transmitted light, and it seems unlikely that this should not also be the case with opaque mineral optical figures obtained with reflected light. The effects of rotating the objective and the use of the rotating adapter on thin section pleochroism and birefringence were not investigated, but it seems probable that these properties, like the interference figures, would suffer no change.

However, the rotating adapter, by changing the tube length of the microscope, does have an effect as it changes the size of the "optic angle." The changes caused are not consistent; with some objectives the "optic angle" is increased by a large amount and with other objectives by only a small amount, if at all. Since similar changes are noted when examining the same optical figure with different objectives without the adapter, this

effect of the rotating adapter seems of little importance in an explanation of the phenomenon under discussion.

The different "optic angles" obtained with different objectives without the adapter vary in a narrow range of 0–10° from the mean value for each mineral. As the variations noted are not consistent with an increase or decrease in tube length of the microscope, it may be that they are due in part to differences in the converging powers of the objectives.

Of 16 objectives used in examining the optical figures from six standard opaque mineral crystals, showing types II, III, and IV, all but two gave consistent results. These objectives range in date of manufacture from the 19th century to 1947, and include the products of Bausch & Lomb, Leitz Wetzlar, and Spencer. The two exceptions, both old objectives, showed the optical figures exactly inverted, or rotated 90° from the optical figures obtained with the other objectives. This may be due to the phenomenon described above, or, perhaps, it may be due to the optics of the objectives.

Other than the major problem of the phenomenon itself, there are three related minor problems. The first of these is the observation that most objectives yield the same kind of figure for each mineral, which has been examined with more than one objective. Allied to this observation is the unusual, though not perfect, agreement in the determinations of such features as rotation colors by different workers. No comment is offered at present toward a solution of this problem.

The second problem is the observation that all objectives, when rotated show the same phenomenon on pyrite, but differ from this in the changes caused on covellite and stibnite. Apparently the changes caused on covellite and stibnite are, to some extent, governed by the nature of the figure; the changes are not exactly equivalent to a superposition of the pyrite reaction on the figures of these minerals. This seems a likely conclusion, when it is considered that covellite and stibnite are anisotropic, while pyrite is isotropic; the different optical constants of anisotropic minerals are of varying importance according to the orientation of the optical directions with respect to the planes of polarization of the polarizer and analyzer. This explanation suffers from the observation that different objectives, when rotated, do not cause the same changes in the optical figures on covellite and stibnite. This problem requires further investigation.

With types II and IV figures the separation of the isogyres for any particular orientation of the mineral is a maximum separation for that orientation, which is not exceeded on rotating the objective through 360°. No explanation of this observation is offered. Obviously this phenomenon is related to the type of figure, and is governed by the orienta-

tion of the mineral. Here, again, because this was noted on types II and IV figures, and because the different objectives, when rotated, did not cause the same effect, further investigation is necessary. In fact this difference in behavior of different objectives on types II and IV figures has a direct bearing on the solution of the major problem at hand, and, in itself, poses a problem of no minor importance.

Other than these statements, no explanations are offered for these phenomena. In fact this article is presented for publication primarily to bring these observations to the attention of mineralogists, to indicate some of the interesting and noteworthy results of an investigation into opaque mineral optical figures, and to invite discussion of this phenomenon.

The author wishes to express his appreciation for the kind help and suggestions given him by Bausch & Lomb Optical Company and Mr. G. T. Hammond. Dr. George Tunell's interest and suggestions are gratefully acknowledged.

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ADDITIONAL DATA ON THE PROPERTIES OF BECQUERELITE AND BILLIETITE

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ABSTRACT

The principal properties of becquerelite are restated. Observations made on billietite have led the authors to consider it as a variety of becquerelite.

BECQUERELITE. It may be useful to recall here some data concerning that mineral. One of us has described the properties of becquerelite in the first publication on this species as follows (1), (2): Translucent, brownish yellow mineral, coating pitchblende; easily cleavable; chemical tests give uranium, lead, and water; good crystals are rare; generally pseudohexagonal plates parallel to $c(001)$, showing under the microscope a good interference figure and negative acute bisectrix perpendicular to $c(001)$. There is another cleavage parallel to $m(110)$; the angle $m(110):m(\bar{1}\bar{1}0) = 62^\circ$; twinned on plane (110). Thin cleavage plates [$c(001)$] show dichroism in yellow shades. A chemical analysis on 0.3173 gr. of material was made and gave the following results:

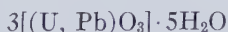
H ₂ O.....	10.02%
PbO.....	5.38
UO ₃	82.73

Later we discovered becquerelite crystals with a prismatic habit and always elongated parallel to the b axis; they did not contain lead. In addition to the perfect cleavage $c(001)$, three other cleavages can be observed on these crystals: $m(110)$, $d(101)$ and $b(010)$.

An analysis of these crystals gave the following (3):

H ₂ O.....	9.91%
UO ₃	90.09

In Fig. 1 are shown several habits of becquerelite crystals as seen with the microscope. The twins (a), (b) and (c) are becquerelite crystals containing lead; no lead was detected in crystals of type (d). Using the cell dimensions and gravity obtained on prismatic crystals, V. Billiet and W. F. de Jong (3) have calculated for becquerelite the formula: $2\text{UO}_3 \cdot 3\text{H}_2\text{O}$. They found 13 molecules in the unit cell which seems unlikely. H. Brasseur (5) recently proposed another formula, namely $3\text{UO}_3 \cdot 5\text{H}_2\text{O}$ with 8 molecules in the unit cell. Assuming this formula to be correct the formula for becquerelite containing lead, becomes



where lead appears to replace a small amount of uranium.

Certain differences were noted in the optical properties of both varieties of the mineral. For instance, the indices of refraction of the lead bearing becquerelite, as determined by Billiet (6) are: $\alpha=1.75$, $\beta=1.87$, $\gamma=1.88$. As previously stated, thin cleavage plates parallel to $c(001)$ are dichroic; moreover the angle of the optic axes, which is $2V=30^{\circ} 35'$ becomes 0° at a temperature of 100° C.; this can be observed on crystals embedded in Canada balsam.

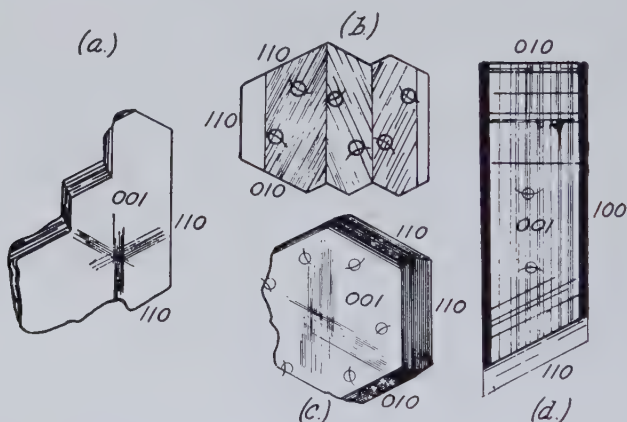


FIG. 1. (a), (b), and (c) are becquerelite twins; (d) is a crystal showing the traces of the cleavages (010), (101), and (110).

The indices of refraction of the becquerelite, $3\text{UO}_3 \cdot 5\text{H}_2\text{O}$, are lower than those mentioned above because of the absence of lead; we found: $\alpha=1.725$, $\beta=1.825$, $\gamma=1.83$; these values are in close agreement with the indices of becquerelite published by the U. S. Geological Survey (7): $\alpha=1.735$; $\beta=1.82$; $\gamma=1.83$. It is to be noted, that the cleavage plates [$c(001)$] of this variety of becquerelite are not dichroic and that the optic angle is not altered by an increase in temperature for crystals embedded in Canada balsam and heated up to 100° C.

BILLIETITE. J. F. Vaes (8) has given the name of billietite to an uranium mineral from the Shinkolobwe mine in Katanga. According to the author, billietite is an hydrated barium uranate; its precise chemical formula has not yet been established as only microchemical tests were made. The presence of barium in the mineral has been confirmed by spectral analysis. Billietite contains no lead. Crystals range from $\frac{1}{2}$ to 2 mm. and closely resemble becquerelite; they are orthorhombic with tabular development, translucent, and amber yellow in color.

Vaes gives the following symbols to the observed forms: (010); (110); (101); (011) and (111); the value of the angle (101): $(\bar{1}01)$ is $61\frac{1}{2}^{\circ}$. There

is a perfect cleavage parallel to (010); twins were noted parallel to (111) and (101). The acute bisectrix is perpendicular to (010); $2V = \pm 36^\circ$; $r > v$; dispersion strong; the axial plane is parallel to (001). Vaes found that the refractive indices are very near to those of becquerelite but does not mention their values. Dichroism is apparent in cleavage plates parallel to (010) in various shades of yellow; $Z = a$; $Y = c$; $X = b$.

New observations. We have found flat, tabular pseudohexagonal crystals of billietite (Fig. 2) associated with soddyite and coating a fragment

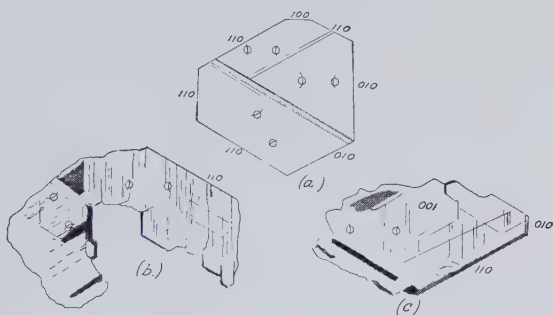


FIG. 2. Twinned crystals of billietite lying on perfect cleavage plane (001). (a) is very regular and does not show any other cleavage; (b) and (c) show distinctly the traces of two different cleavages (110) and (010).

of uranium ore consisting of soddyite and curite. In order to emphasize the strong crystallographic similarity of billietite and becquerelite, we think it advisable to interchange the axes b and c of billietite. We are giving, below, the indices of the forms in the new orientation, opposite to those proposed by Vaes.

FORMS OF BILLIETITE CRYSTALS

J. F. Vaes	New position (Schoep & Stradiot)
(010)	c (001)
(101)	m (110)
(011)	d (101)
(110)	e (011)
(111)	p (111)

The indices of the perfect cleavage plate becomes $c(001)$; $61\frac{1}{2}^\circ$ is the angle between $m(110)$ and $m(\bar{1}\bar{1}0)$; the plane of the optical axes is parallel to $a(100)$, and the acute bisectrix is perpendicular to $c(001)$. We have measured the indices of refraction using appropriate solutions of phosphorus in methylene iodide, and we find: $\alpha = 1.73$; $\beta = 1.82$; $\gamma = 1.83$.

All the data on billietite and becquerelite are grouped in the following table.

	Becquerelite	Becquerelite	Billietite
Chemical composition	$3[(U, Pb)O_3]5H_2O$	$3UO_3 \cdot 5H_2O$	UO_3, Ba, H_2O
Crystal system	orthorhombic	orthorhombic	orthorhombic
Twinning	(110)	not observed	(110) and (111)
Cleavages	$c(001), m(110), d(101), b(010)$	$c(001), m(110), d(101), b(010)$	$c(001), n(110), b(010)$
Color	amber yellow translucent	amber yellow translucent	amber yellow translucent
Pleochroism	in yellow shades $X = \text{colorless}$ $Y = \text{yellow}$ $Z = \text{deep yellow}$	in yellow shades $X = \text{colorless}$ $Y = Z = \text{yellow}$	in yellow shades $X = \text{colorless}$ $Y = \text{yellow}$ $Z = \text{deep yellow}$
Optic. Orient.	$X = c, Y = a, Z = b$	$X = c, Y = a, Z = b$	$X = c, Y = a, Z = b$
Acute bissect.	perpend. to $c(001)$	perpend. to $c(001)$	perpend. to $c(001)$
Axial plane	parallel to $a(100)$	parallel to $a(100)$	parallel to $a(100)$
2V	$\pm 30^\circ$	$\pm 30^\circ$	$\pm 36^\circ$
Indexes of refraction	$\alpha = 1.75$ $\beta = 1.87$ $\gamma = 1.88$	$\alpha = 1.725$ $\beta = 1.82$ $\gamma = 1.83$	$\alpha = 1.73$ $\beta = 1.82$ $\gamma = 1.83$

CONCLUSION

Apart from the presence of barium, there is scarcely any difference between billietite and the becquerelite that does not contain lead; there is more difference between the two types of becquerelite. It seems very likely, therefore, that billietite is merely a variety of becquerelite containing some barium substituting for uranium. The problem can not be considered solved until analyses and x-ray study are made. This is one more example showing that new names for minerals should not be proposed on the basis of incomplete data.

On first inspection there seemed to be a relationship between the pseudohexagonal tabular habit of becquerelite and the presence of Pb or Ba in the molecule; it is a fact that we never found pseudohexagonal tabular becquerelite crystals that did not give a reaction for Pb or Ba;

but it must be said that some prismatic crystals of becquerelite have the same indices of refraction as plumbobecquerelite.

The presence of Pb or Ba in the becquerelite molecule is perhaps to be explained as resulting from the replacement of U^{+6} by Pb^{+2} or by Ba^{+2} ; the ionic radii of those elements have nearly the same value; but we do not understand why such replacements do not occur in other uranium hydroxides, for instance in schoepite and in paraschoepite.

A cleavage parallel to (101) has not yet been observed on billietite, perhaps because of the insufficient number of crystals examined.

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NOTES AND NEWS

MINERAL DETERMINATION BY MEANS OF PUNCHED CARDS

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To the beginner in mineralogy the determination of minerals is a difficult and perplexing problem. He is taught how to make certain observations and determinations such as luster, hardness, specific gravity, and streak, but only through experience can he integrate these observations in a manner that will enable him to quickly determine the unknown mineral. To aid in such identification, determinative tables have been devised and published separately or appended to text books of elementary mineralogy.

The chief difficulty of all such tables is that the various properties must be considered in a prescribed order. For example, a common sequence is luster, hardness, cleavage, specific gravity. If the outstanding property is a high specific gravity, it can not be used until the mineral is classified first by several other less distinctive and perhaps less reliable properties.

In an effort to overcome this shortcoming of the ordinary determinative table, Donnay* in 1938 devised a set of cards or "grids" for the determination of 450 nonopaque minerals. To quote from Donnay's paper, "... a grid is a sheet of cardboard, about 7 by 3 inches on which 45 vertical columns of 10 figures each (0-9) are printed, the columns being numbered from 1 to 45 along the bottom edge of the card. Every one of the 450 figures stands for a mineral that can be looked up in the index." Each card, of which there are approximately 250, represents a property and in it holes are punched in the appropriate places representing all the minerals having that property. For example, there are cards for hardness greater than 1, greater than $1\frac{1}{2}$, greater than 2, etc., and others for hardness less than 1, less than $1\frac{1}{2}$, less than 2, etc. There are similar cards for specific gravity, cleavage, color, crystal system, crystal habit, optical properties, chemical elements, and others. Suppose the properties of a mineral have been determined as: white, H. greater than 5 but less than 7, G. greater than 2.5 but less than 2.9, tetragonal crystals, and no distinct cleavage. The seven cards representing white, $H.>5$, $H.<7$, $G.>2.5$, $G.<2.9$, tetragonal, and no distinct cleavage would be located in the pack, withdrawn, and placed over each other. If only one hole is common to all the cards, it represents the unknown mineral, the name of which can be looked up in the index. If more than

* Donnay, J. D. H., A small set of grids for the determination of non-opaque minerals: *Am. Mineral.*, **23**, 91-100 (1938).

one hole is common to all the cards, additional cards representing other properties must be withdrawn and placed over the others.

The new type of punched cards described below differs from Donnay's in many respects but notably in that each card represents a mineral and not a property. Thus as many minerals may be considered as one wishes to add cards. The cards† are $7\frac{1}{2}$ inches by $3\frac{1}{4}$ inches and have $76\frac{1}{8}$ -inch holes punched around the border—28 holes at the top, 30 at the bottom, and 9 at each side (Fig. 1). Each hole, the same hole on all the cards, has been assigned a property, the name of which is printed adjacent to it.

FIG. 1. Mineral identification card of apatite.

The properties and the divisions of them are:

Luster

Metallic
Nonmetallic

Color

Red
Orange
Yellow
Green
Blue
Violet
Brown
Black
White or colorless
Silver
Bronze

Crystal System

Isometric
Tetragonal

Hexagonal
Orthorhombic
Monoclinic
Triclinic

Streak

Red
Orange
Yellow
Brown
Black
Colorless

Specific Gravity

Less than 2
2.0-2.39
2.4-2.79
2.8-3.39
3.4-3.99
4.0-4.99

† The cards, sold under the name of "Keysort," are manufactured by the McBee Company, Athens, Ohio.

5.0-5.99	<i>Cleavage</i>
6.0-6.99	Show cleavage
7.0 and greater	One direction
	Two directions
	Three or more directions
<i>Hardness</i>	
Less than $2\frac{1}{2}$	
$2\frac{1}{2}$ to less than 3	
3 to less than $5\frac{1}{2}$	
$5\frac{1}{2}$ to less than 7	<i>Fusibility</i>
7 and greater	Fusible at 1

In addition, 27 of the commonest elements as well as CO_2 , SO_4 , and H_2O are represented by the holes at the bottom of the card.

The card representing any given mineral has had V-shaped notches punched in such a way that the holes representing the properties of that mineral are opened to the edge. As an example, consider the apatite card shown in Fig. 1. Notches are punched for: *nonmetallic*; the various colors that it might show—*green, violet, brown, colorless*; *hexagonal*, crystal system; *colorless* streak; specific gravity 2.8–3.39; hardness 3– $5\frac{1}{2}$; chemical elements *Ca, Cl, F*, and *P*. In the sets thus far made there are 150 cards representing 150 of the commonest minerals, those usually considered in an elementary course in mineralogy. The number of cards could be increased easily if one wished to consider a larger number of minerals.

In using the cards for determination, they are stacked together evenly with the clipped corners at the upper right. A slender metal rod is then pushed through the hole in the cards representing the property to be considered. The first separation can be made according to any property, but it is usually convenient to make it on the basis of luster (metallic or non-metallic), for this immediately eliminates a large number of minerals. For submetallic minerals, both metallic and nonmetallic are punched.

If apatite is the unknown mineral and its properties have been determined by the student, the rod is put in the hole "nonmetallic." By raising the rod all the cards punched for nonmetallic drop out of the pack and the apatite card would be among them. The next separation might be on the basis of color. If the apatite is brown, the needle is inserted in the "brown" hole and all the cards representing minerals that may be of this color are separated from the others. Thus it would be found that 37 minerals of the 150 are nonmetallic and may be brown. Another separation on the basis of hardness (H. greater than 3 to less than $5\frac{1}{2}$) leaves 16 cards, among which is the one representing the unknown mineral. A further separation on the basis of specific gravity (G. 2.8–3.39) leaves only 3 cards—collophanite, fluorite, and apatite. If the apatite were in crystals, a last separation on the basis of hexagonal crystal system would leave *apatite* as the only mineral satisfying all the requirements.

It may happen that the easily observed properties will not isolate a card but two or three cards are left. In such a case it is easy for the student to look up the minerals in question and make an additional test to positively identify his mineral.

After a determination has been made, it is unnecessary to return the cards to any given place in the pack for, stacked together in any order, they are ready for use again.

In teaching, this method of mineral identification has an advantage over ordinary tables, for when the card for a given mineral is isolated, the student has before him more than a name. By observing the notches he can see the ranges within which the hardness and specific gravity fall and can note the colors, the crystal system, cleavage, and the chemical elements. More important than these would be the notes made on the card by the student pointing out tests that can be used to distinguish between similar appearing minerals.

The two holes at the extreme left at the top of the cards have not been used. Thus, if the student wishes to make use of other properties, they can be assigned to these holes and the notches made with scissors. The number of minerals is by no means fixed. The 150 included is about the maximum number studied in most elementary courses of mineralogy. If certain instructors wish to include others, this can easily be done by notching unpunched cards at the appropriate places and writing the mineral names at the center. Five blank cards are included with each set for this purpose.

Unlike Donnay's grids which consider 450 nonopaque minerals, this set of punched cards* is designed for the student of elementary mineralogy and takes into account only those properties most easily determined. With these limited properties it would undoubtedly be impractical to consider more than 250 minerals. However, the method would lend itself to a much more comprehensive adaptation. Larger cards with double rows of holes are available and on them it would be possible to include such additional data as refractive indices, 2V, extinction angle, dimensions of the unit cell, and d spacings as obtained from x -ray powder photographs. In fact after seeing the set of 150 small cards, Mr. E. E. Fairbanks obtained some larger cards and punched a few sets for use in the determination of the opaque minerals.†

This type of punched card for mineral identification has several advantages over determinative tables: (1) The properties can be consid-

* The set of 150 cards can be purchased from Ward's Natural Science Establishment, Inc., Rochester, N. Y.

† Fairbanks, Ernest E., The punched card identification of ore minerals: *Econ. Geology*, **41**, 761-768 (1946).

ered in any order; (2) all the cards representing minerals containing a given element or belonging to a given crystal system can be quickly separated and studied as a group; (3) there is ample room for making notes on both the face and back of the card for future reference; (4) the number of minerals considered is flexible. More cards can be added if necessary or cards representing minerals not studied in a given course can be removed, thus simplifying the determinative procedure.

The greatest advantage these punched cards have over Donnay's grids is that the separation by properties is easily and quickly made and it is unnecessary to thumb through the pack looking for the card representing a given property. Moreover, after a determination is made, the cards can be stacked together in any order and without refiling are ready for use again.

FIRE HAZARD WITH C. D. WEST'S HIGH REFRACTIVE INDEX LIQUIDS*

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Twelve years after C. D. West¹ proposed the use of yellow phosphorus-sulfur-methylene iodide solutions, there have been few published data concerning their actual use. Recently, a discussion by Bruun and Barth² emphasized the stability of these liquids, with special reference to the refractive index, over a period of five years.

West very properly mentions that, "the liquids are stored in glass-stoppered bottles which are kept in a covered metal container, the latter to reduce fire hazard and to exclude light which has an adverse effect. In the present experiments, a half inch layer of water was kept over the liquids." It might be thought that such precautions would ensure safety; yet, under these very conditions, there is still a very serious danger of fire from this treacherous preparation.

A set of six liquids, prepared a year ago, was contained in 35 ml. glass bottles, each with ground-in glass applicator rod, and ground-in glass cap. The liquid was covered with about an inch of water. Finally, the six bottles were placed in a galvanized iron box, with a fairly close-fitting lid.

At intervals of several months throughout the year, the liquids were inspected and found to remain clear and apparently unaltered. What was not noted, however, was the gradual evaporation of the water layer in some of the bottles. Concerning this water layer, West observes, "The stability of the liquids on storage without a water layer has not been de-

* Published with permission of the Director, U. S. Geological Survey.

¹ West, C. D., Immersion liquids of high refractive index: *Am. Mineral.*, **21**, 245-249 (1936).

² Bruun, Brynjolf, and Barth, Tom. F. W., Stability on storage of the high refractive index liquids of C. D. West: *Am. Mineral.*, **32**, 92-93 (1947).

terminated; it could hardly be less than what has been indicated by the foregoing," i.e. substantial stability over $9\frac{1}{2}$ months. The presence of this water layer is, however, an extremely important matter.

Recently, the metal box was opened and immediately white fumes of phosphorus pentoxide were observed, with the characteristic garlic odor, and, almost simultaneously, a flash of flame. The metal box was heavily corroded and a yellow deposit—presumably phosphorus and sulfur—had formed around the neck of some of the bottles.

The explanation of these phenomena is that during the year the water had evaporated, notwithstanding the double ground-in seal of the containing bottle. Once the protective water was gone, the phosphorus-sulfur-methylene iodide "crept" up the interior of the bottle, and finally outside, whereupon the solvent iodide evaporated leaving a dangerous deposit of phosphorus-sulfur outside of the bottle. In contact with wood or combustible matter, a dangerous fire could have ensued. The metal box suggested by West is some protection, but has the disadvantage of hiding possible evaporation of the water layer.

Glycerine has been suggested as better than water for preventing evaporation of the phosphorus liquid (Dr. John C. Rabbitt, oral communication). In any case, a layer of protective liquid should always be maintained, with frequent inspections to ensure its presence. Especially, phosphorus liquids should never be put in "dead" storage in wooden cupboards!

CRYSTALS OF PARASCHOEPITE¹

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Four of our best paraschoepite crystals, although very small, could be measured; their habit is short prismatic (Fig. 1). Results are given below:

	ϕ	ρ	
<i>c</i> (001)	—	0°	Observed on all crystals.
<i>b</i> (010)	0°	90°	Observed on all crystals.
<i>a</i> (100)	90°	90°	Observed on all crystals.
<i>m</i> (110)	66°59'	90°	Observed on all crystals.
<i>d</i> (011)	0°	41°04'	Observed on two crystals.
<i>f</i> (021)	0°	60°27'	Observed on all crystals.
<i>e</i> (041)	0°	72°19'	Observed on one crystal; face of poor quality.
<i>p</i> (111)	67°46'	66°26'	Faces of this form on one of the crystals are of poor quality.
<i>q</i> (124)	49°59'	33°47'	Observed on three crystals.
<i>o</i> (122)	49°47'	53°27'	Observed on two crystals.

¹ *Am. Mineral.*, **32**, 344-350 (1947).

The small differences between the values of ϕ and ρ found for paraschoepite and for schoepite crystals² are only due to the fact that, generally speaking, the crystals of paraschoepite are not of the same good quality as those of schoepite. It seems justifiable to conclude that there is no difference whatever between the parameters of both species.

To avoid any mistake we have determined the indices of refraction of each paraschoepite crystal measured. Fig. 1 represents the habit of all crystals of paraschoepite measured by us. Microscopic crystals of the mineral are often elongated parallel to the c -axis (Fig. 2).

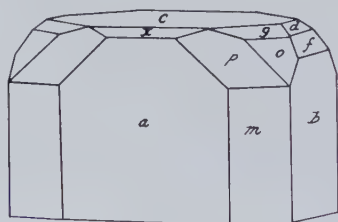


FIG. 1. Crystal of paraschoepite



FIG. 2. Crystal of paraschoepite elongated parallel to the c axis.

ACTA CRYSTALLOGRAPHICA, VOL. 1. PART 1

The first issue of *Acta Crystallographica* has been awaited with interest by crystallographers, mineralogists, chemists, physicists, and other scientists the world over. It was published in March of this year for the International Union of Crystallography by the Cambridge University Press, 200 Euston Road, London, N.W. 1, England. The editor is P. P. Ewald, of The Queen's University, Belfast, Northern Ireland, and co-editors include R. C. Evans of the Crystallographic Laboratory, Caven-

² Palache, C., Crystallography of the uranium oxides: *Am. Mineral.*, **19**, 309 (1934).

dish Laboratory, Cambridge, England; I. Fankuchen, Polytechnic Institute of Brooklyn, 99 Livingston St., Brooklyn 2, N. Y.; A. V. Shubnikov, Institute of Crystallography, Academy of Sciences of the U.S.S.R., Starometny 35, Moscow 17, U.S.S.R.; and J. Wyart, Laboratoire de Minéralogie à la Sorbonne, 1 rue Victor-Cousin, Paris V, France. Manuscripts will be accepted in English, French, German, and Russian. Those in English should be sent to R. C. Evans or I. Fankuchen for consideration; in French to J. Wyart; in German to P. P. Ewald; and in Russian to A. V. Shubnikov. In addition to this staff of able editors, on the advisory board are listed the following men who are world known for their significant contributions to crystallography: Sir Lawrence Bragg, M. von Laue, C. Mauguin, P. Niggli, L. Pauling, and R. W. G. Wyckoff. This is truly an impressive array of godfathers, and their support alone should go far to insure the initial success of the undertaking.

The first issue sets a high standard, both in context and format. It contains 48 pages divided among six main articles, three shorter contributions, notes and news, and book reviews. Leading articles are by J. Garrido (Spain) on the diffuse scattering of x -rays by NaClO_3 crystals; by C. J. B. Clews and W. Cochran (England) on the structures of two pyrimidines; by K. Lonsdale (England) on the diffuse scattering of x -rays by single crystals; by B. Jerslev (Sweden) on the structures of two hydroxylammonium halides; by H. G. F. Winkler (Germany) on the synthesis and structure of eucryptite; and by W. L. Roth and D. Harker (U. S. A.) on the structure of octamethylspiro [5.5] pentasiloxane. Brief abstracts in English precede all the articles.

The journal is in imperial quarto size with two columns to the page. The paper is of good quality and the print is easy to read. Line drawings and photographs are uniformly excellent and the binding is stitched. The price for the six yearly issues is \$10 or £2.10s. Although it is stated that, "It is hoped that this moderate price will ensure a large number of subscribers," the price seems high in comparison to American journals on related subjects, who offer their subscribers much larger or more numerous issues at a similar standard of quality, generally at a lower price. It is believed that the cost may deter many young crystallographers from subscribing, whereas a more modest price might make it available to the graduate student who is already beset on all sides by the increased costs of education and living.

Certainly the publication of this journal comes at an opportune time, when interest in crystallography is rising rapidly. The growing concern with crystallographic techniques is manifest not only from the vigorous activity of various societies (for example, the Crystallographic Society of America, American Society for X-ray and Electron Diffraction), but also

from the lengthy list of commercial firms whose financial support has helped to bring about the birth of this journal. Undoubtedly the time will come when the science of crystallography will be represented in universities as a single curriculum devoted to the training of students of atomic structure, be it of minerals, or of organic or inorganic chemicals. The journal may be ordered in the United States through The American Institute of Physics, 57 East 55th Street, New York 22, N. Y.

E. WM. HEINRICH, *University of Michigan*

ANATOLII KAPITONOVICH BOLDYREV*

1883-1946

INNA V. POIRÉ

The outstanding Russian explorer and scientist, Anatolii Kapitonovich Boldyrev, was killed on March 24, 1946, in an automobile accident near Magadan in Eastern Siberia.

A. K. Boldyrev was born in 1883 in the Ukraine. When he graduated from the Mining Institute in St. Petersburg in 1920, he had attained recognition as a scientist and was appointed Professor of crystallography and mineralogy at the Institute. He also organized and directed until 1937 the E. S. Fedorov Institute of Crystallography and the laboratory for the study of minerals by x -rays, where he introduced new methods of research. He was the force of the Institute and a real friend and mentor of his students. Boldyrev studied minerals in nature, tried to determine substances on the basis of their crystalline structure, and gave mathematical arguments to his research. He applied this study to mineral resources and to their utilization. In 1934, when scientific degrees were restored in the USSR, Boldyrev received a doctorate degree on the basis of his scientific activity and publications. His 85 published papers and unpublished manuscripts cover a variety of subjects—crystallography, mineralogy, petrology, ore deposits, mineral resources, and hydrology.

Boldyrev was simple, friendly, modest, self-possessed and reserved. Although his scientific career was interrupted many times by political upheavals, his accomplishments were numerous, substantial and lasting. With his passing, Russian science has lost a remarkable man, scientist, and teacher.

Dr. Alfred C. Lane, formerly Pearson professor of geology and mineralogy at Tufts College, died April 15 in Cambridge, Massachusetts. At one time Dr. Lane was head of the National Research Council's committee on estimation of geologic time by atomic disintegration and was president of the Geological Society of America in 1931.

Dr. Lewis G. Westgate, emeritus professor of geology at Ohio Wesleyan University, died March 30 at his home in Delaware, Ohio, at the age of seventy-nine. He had been a professor at the University from 1900 until his retirement in 1939 and resumed his duties from 1942 to 1944.

Geologists and mineralogists will be interested in a recent publication of the Colorado School of Mines entitled "Guide to the Geology of Central Colorado" (Vol. 43, No. 2, of the School's *Quarterly*), which was issued as the guidebook for the three field trips of the 33rd annual meeting of the American Association of Petroleum Geologists held

* Published by permission of the Director, U. S. Geological Survey.

in Denver in April. The publication is profusely illustrated with figures, plates, and a 24"×28" index map of the state, showing major geologic structure and significant oil and gas information. Well-known geologists from the consulting and educational fields and from the U. S. Geological Survey have contributed authoritative articles, and in addition there are discussions of the areas in which the field trips were made. The guidebook, which may be obtained from the Department of Publications, Colorado School of Mines, Golden, is priced at \$3.00 (postpaid).

Bulletin 848, *The Microscopic determination of the nonopaque minerals* (2nd edition) by E. S. Larsen and Harry Berman, 1934 [1948], VI, 266 pages, 7 figures, has been reprinted and is offered for sale by the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Price 50 cents.

NOMINATIONS FOR OFFICERS OF THE MINERALOGICAL SOCIETY OF AMERICA FOR 1949

The Council has nominated the following as officers of the Mineralogical Society of America for the year 1949 to be voted on by the membership at the November election:

President: John W. Gruner, University of Minnesota, Minneapolis, Minnesota.

Vice-President: J. D. H. Donnay, The Johns Hopkins University, Baltimore, Maryland.

Secretary: C. S. Hurlbut, Jr., Harvard University, Cambridge, Massachusetts.

Treasurer: Earl Ingerson, U. S. Geological Survey, Washington, D. C.

Editor: Walter F. Hunt, University of Michigan, Ann Arbor, Michigan.

Councilor (1949-52): Lewis S. Ramsdell, University of Michigan, Ann Arbor, Michigan.

ANNOUNCEMENT OF THE TWENTY-NINTH ANNUAL MEETING

The twenty-ninth annual meeting of the Society will be held at the Hotel Pennsylvania in New York City, November 11-13, 1948, in connection with the sixtieth annual meeting of the Geological Society of America.

Members of the Society who are planning to present papers at the scientific sessions of the annual meeting should notify the Secretary as soon as possible in order to receive the proper blanks for their abstracts. All abstracts must be in the Secretary's office by *September 1*. By a ruling of the Council no abstracts will be accepted for presentation by title only.

Advance announcement of the annual meeting will be distributed to members of the Society, with the ballot for officers, in September. The final program of the meeting, including the schedule of papers, abstracts, and other information will be sent to each member about November 1st. Further specific information regarding the annual meeting may be obtained from the Secretary's office.

C. S. HURLBUT, JR., *Secretary*

BOOK REVIEW

ERUPTIVE ROCKS, their genesis, composition, classification, and their relation to ore-deposits with a chapter on meteorites, by S. JAMES SHAND, third edition, Thomas Murby & Co., 40 Museum Street, London; John Wiley & Sons, Inc., 440 Fourth Ave., New York, 1947. xvi+488 pages. Price \$7.50.

The first edition of this excellent book was published in 1927 and the second in 1945. In the third edition the chapters have been rearranged and the text somewhat revised, but

the most important change is the insertion of three chapters "devoted to a discussion of the link between eruptive rocks and certain types of ore deposits." The headings of these chapters are:

Chapter X Late-magmatic and post-magmatic reactions

XI The genesis of pegmatites

XII Eruptive rocks and ore deposits

The three chapters give briefly the carefully considered views of a petrologist on this subject and should be read by every geologist interested in ore deposits. Shand believes that on crystallization of the magma beyond a certain stage it will liberate the excess of water as a second fluid phase and that in a deep-seated magma this fluid phase will in all probability be a liquid below its critical temperature. The suggestion that we discard the term pneumatolitic should be followed. It is refreshing to find that Shand believes that the aplites did not crystallize from a dry melt but that they owe their texture to some physical cause. The newer data on the layered pegmatites—such as some of those mined for muscovite and feldspar—are not discussed.

The presentation is concise, the descriptions are clear, the arguments are fair and the conclusions generally convincing. The reviewer is annoyed at the terms "acid granite" and "basic plagioclase." The author will agree that no granite is acid and no plagioclase basic. "Silicic" and "calcic" describe the rock and the feldspar much better.

The author wisely avoids lists of criteria for the determination of some fact such as the order of crystallization of the minerals in rocks, recognizing that such lists of rules are commonly unreliable.

Shand's classification of igneous rocks is logical and quantitative. He has two groups based on the crystallinity, five on degree of saturation with silica, four on the content of alumina, four on the composition of the feldspar and four on the color index. Most petrographers accustomed to some modification of the Rosenbusch classification will probably see no great advantage in giving so great importance to alumina.

Shand's book is the best book in English and probably in any language on modern igneous petrology. It is an excellent textbook and should be interesting and profitable reading for anyone who is interested in petrology.

ESPER S. LARSEN, JR.,
Harvard University

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